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# Evaluation of Adhesive Binders for the Preservation of In-Situ Aboriginal Surface Finishes at Mesa Verde National Park

Rebecca J. Carr  
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# Evaluation of Adhesive Binders for the Preservation of In-Situ Aboriginal Surface Finishes at Mesa Verde National Park

## **Disciplines**

Historic Preservation and Conservation

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EVALUATION OF ADHESIVE BINDERS FOR THE PRESERVATION OF  
IN-SITU ABORIGINAL SURFACE FINISHES AT  
MESA VERDE NATIONAL PARK

Rebecca J. Carr

A THESIS

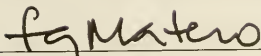
in

Historic Preservation


Presented to the Facilities of the University of Pennsylvania  
in Partial Fulfillment of the Requirements for the Degree of

MASTER OF SCIENCE

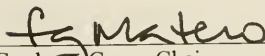
2002



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## Chapter 1

### INTRODUCTION

Archaeologists attribute the architecture of Mesa Verde National Park to Ancestral Puebloan people who constructed and inhabited these structures prior to A.D. 1300. At this time, sandstone masonry structures coated with earthen plasters were constructed at more than 600 sites within the modern day boundaries of Mesa Verde National Park.

Mesa Verde contains a high percentage of its original earthen surface finishes. These earthen renderings are interpreted to the public on a daily basis. Earthen surface finishes contribute to both the informational value contained within a site and to the durability of these structures. At Mesa Verde, surface finishes were originally a part of building maintenance as well as a means for artistic, social and cultural expression. Today, prehistoric surface finishes play an important role in the maintenance and interpretation of these archaeological sites. Earthen finishes serve as a sacrificial layer applied to extend the life of an underlying masonry substrate and in doing so, these layers retain an interpretive value. Hand prints, anthropomorphic figures and geometric shapes decorate the walls of many rooms. Pictographs, color schemes, and analysis of finish layers contribute interpretive data regarding the use and history of Ancestral Puebloan architecture.

Earthen surface finishes at Mesa Verde are an extremely important resource for archaeologists, scientists, managers, planners and the park's culturally affiliated tribes.



These painted designs hold significance for the modern Pueblo people and should be preserved for the future generations of all Americans.

As a National Park, Mesa Verde has been set aside as an important resource for future generations. It has been designated as a World Heritage Site, worthy of preservation, research and interpretation. As such, it is important to select methods of site stabilization that will prolong the life of this invaluable resource and enable us to conduct future research that will enhance our understanding of the materials used to construct these structures. Aside from tourism, it is the responsibility of the National Park Service to maintain these structures in their current state. Every year, fragments of earthen plaster detach from the walls. It is our responsibility to future generations to retain as much of this cultural resource as possible.

Between 1994 and 1997, a documentation and treatment program for Earthen Surface Finishes was developed at Mug House in Mesa Verde National Park. A partnership between the University of Pennsylvania Architectural Conservation Lab and the National Park Service has produced a vast quantity of research regarding the mechanisms of surface-finish deterioration, low-impact methods for stabilization, and detailed documentation. Consultation with Native American tribes led researchers at the university to seek a natural adhesive as an alternate to the popular acrylics used to reattach and consolidate earthen surface finishes.<sup>1</sup>

The University of Pennsylvania Architectural Conservation Lab designed and implemented a documentation method to record and monitor each of the factors that





contributes to a loss of earthen surface finishes at these sites. A total of 20 conditions terms were identified and each is documented on site before any treatments are conducted.

The university also developed treatment methods to stabilize earthen surface finishes that are delaminated and detached from their substrate. One such treatment uses either of two gelatin solutions that are diluted with water to form an adhesive solution. This adhesive is injected into exposed voids to stabilize isolated areas of surface finish that are already blistered, delaminated or detached. These damaged finishes are in the most jeopardy of loss since blistering, delamination and detachment are the immediate precursors to the loss of finish layers.

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<sup>1</sup> Personal communication with Frank Matero, University of Pennsylvania Architectural Conservation Laboratory, 7/23/02.



## Chapter 2

### RESEARCH OBJECTIVES

This study seeks to evaluate the various methods of reattachment used at Mesa Verde by reproducing Ancestral Puebloan surface finishes and subjecting them to accelerated environmental conditions. By this process, a limited spectrum of the deterioration currently observed at Mesa Verde National Park was induced. To this purpose, documentary research and a modified model of experimental archeology have been combined with modern methods of materials testing. This study will provide additional information regarding the mechanisms and rate of deterioration currently observed at these archeological sites.

Four adhesive solutions were selected for materials testing. The two gelatin based solutions currently used by the University of Pennsylvania were tested and compared against acrylic emulsions. The data generated by this research provides comparative data for evaluating the effectiveness of the current treatment methods and to provide additional research that will enhance the understanding of the treatments of these deterioration mechanisms.

Soil samples were collected from Mesa Verde National Park to reproduce the prehistoric surface finishes found at archeological sites within the park. The formulation of these reproductions was based on prior research conducted at Mesa Verde. Loosely based on the principle of experimental archaeology, these small-scale reproductions were subjected to artificially induced accelerated weathering. Monitoring and conditions



recording throughout the weathering process enhanced my understanding of the sequence of symptoms and environmentally induced threats that lead to the deterioration of Ancestral Puebloan surface finishes. This knowledge will be used to further the conservation program at Mesa Verde National Park.

## **2.1 Research Objectives**

- Assess the effects of particle-size distribution on the durability of earthen surface finishes
- Assess the affects of accelerated weathering of surface finishes with a crystalline binder of calcium carbonate as compared to surface finishes with a clay binder alone
- Compile laboratory methods from the fields of conservation and materials testing to produce testing recommendations for the further assessment of earthen surface finishes
- Document the progression of accelerated deterioration of earthen surface finishes as a basis for in-situ monitoring of extant surface finishes at Mesa Verde National Park
- Assess the physical properties of stabilization adhesives currently used at Mesa Verde National Park by researching adhesive formulations and materials testing
- Conduct comparative materials testing of four different adhesive formulations
- Provide recommendations for further study of the adhesive reattachment of earthen plasters



## Chapter 3

### PRINCIPLES OF ADHESION

Adhesives, paints, plasters, mortars, fillers and sealants are formulated according to the mechanical properties of their constituents and the ways that each component interacts on a physical and chemical level. The strength and durability of an adhesive formulation is dependent on the physical properties of the adherents that it joins. Many adhesives do not attach to their adherents on a chemical level. Instead, much of the strength of an adhesive bond is dependent upon the ability of an adhesive to physically coat the surfaces to be adhered. When an adhesive and adherents have similar rates of expansion and contraction, when subjected to environmental fluctuations of temperature and relative humidity, their adhesive bond lasts longer.<sup>2</sup> Along the same lines, an adhesive with greater flexibility can move with the expansion and contraction of its adherents resulting in a more durable bond despite the stresses induced by such movement.

Binding of particles together to form a layer of surface finish can be done in one of three ways. Pigment particles can be bound together mechanically and chemically, as is the case for plasters and true frescoes, where the particles are bound by a system of interlocking mineralogical crystal formations of calcite. Pigments can be bound by film-forming polymers that cross-link at the molecular level, known as chemical adhesion, or

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<sup>2</sup> Berger, Gustav A. and William H. Russell. "Conservation of Paintings : Research and Innovations", Archetype Publications: London, 2000.





by monomers and/or polymers that do not cross-link, but are instead held in close proximity to one another by electrostatic forces characterized as Van der Waals forces.

Van der Waals forces are defined as follows “the physical forces of attraction and repulsion existing between molecules and which are responsible for the cohesion of molecular crystals and liquids. The forces stem partly from dipole-dipole, or dipole-induced-dipole interactions; however, even nonpolar molecules and atoms exert a certain attraction on one another. Van der Waals forces act only over relatively short distances, and are proportional to the inverse of the seventh power of the intermolecular distances.”<sup>3</sup>

The deterioration of earthen surface finishes occurs in several different forms of adhesion loss. This loss is experienced at many levels. Loss of adhesion between particles within a single layer of finish constitutes a loss of cohesion and results in a friable surface in need of consolidation. Interfacial loss of adhesion between layers of finish results in “delamination”. Loss of adhesion from the substrate is termed “detachment” and multiple isolated spaces of interfacial detachment accompanied by deformation are called “blistering”. “Finish cracks” are the result of environmentally induced expansion and contraction; when one layer expands more than the adjacent layer to which it is still adhered. Rapid wetting and drying cycles induce differential shrinkage and swelling of In the case of the uppermost layer of surface finish, this movement causes stress between the unrestrained upper surface of the layer and the restrained lower surface that it is still adhered to. The build up of this stress eventually leads to the strain that causes a break in the cohesive strength between particles within the layer. Thus, cracks are due to

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<sup>3</sup> Matt T. Roberts, Don Etherington and Margaret R. Brown. “Bookbinding and the Conservation of books: A Dictionary of Descriptive Terminology”, Stanford University Libraries, 1994.  
<http://palimpsest.stanford.edu/don/don.html>



differential, environmentally induced, expansion and contraction of one layer when it is still adhered to a more rigidly bound structure. The strength of bonds between the particles of sand within the masonry, finish layers, and interfacial attraction have a substantial impact on the rate of earthen surface finish decay.

The differential expansion and contraction that occurs when one layer is more prone to movement than its underlying layer causes stresses at the interface between these two layers. The build up of these stresses eventually causes the strain that results in the cracking and the blistering or buckling of one layer. Fluctuations in surface roughness and surface accumulations of soot or other oily substances provide areas of weakness where adhesion loss is prone to occur. Variations in layer thickness and particle-size distribution also create areas of weakness within the film where cracking is likely to occur.

The amount of surface area that an adhesive is in contact with an adherent depends on the surface tension/viscosity of the adhesive solution, cleanliness of the adherent, the porosity/absorption of the adherent and its texture/surface roughness. The surface tension of an adhesive solution affects the rate that an adhesive flows when applied to an adherent. This property is measured as the viscosity of the solution. When a solution has low surface tension, it is described as a low viscosity solution and is more able to penetrate into the pores of an adherent. In cases like Mesa Verde, low viscosity also allows the adhesive to absorb into the masonry and plaster finishes, creating a thin film around the particles that make up each of these adherents. The ability of an adhesive to coat these particles is referred to as its wettability. Deposits on the surface of an adherent



can reduce the wettability of even a viscous solution especially if the deposit is hydrophobic. Surface texture of adherents also affects the strength of an adhesive bond. Surfaces with measurable surface roughness and high porosity more readily retain both moisture and adhesives. Once an adhesive film has formed, its presence within these spaces provides a mechanical keying between the adhesive and adherent.

### **3.1 Introduction to Earthen Surface Finish Technology**

Within European tradition, an abundance of fine arts paintings and architectural surface coatings were commonly engineered as a three-coat application. These historic plasters consist of an initial scratch coat, a secondary brown coat and a final finish coat. Using historical plaster applications as a model, the thin wash layers with different particle-size distribution might be the final applications of surface finish for a given scheme. Since surface finishes are periodically reapplied and because exposed surface finishes are subject to erosion, a combination of particle-size distribution to determine finish formulation and layer thickness measured on the microscopic level, is required to accurately interpret the sequence of aesthetic and maintenance requirements of a wall. On the broad scale, this will contribute information regarding the occupation sequence of individual rooms.

Layering of surface coatings is a practical solution to increase the durability of such finishes. All materials expand and contract in response to atmospheric changes. Thermal and moisture expansion and contraction vary with the type of material that is subject to these changes. Factors that affect this movement are porosity, pore alignment, rigidity of internal structure, and surface permeability. The exteriors of buildings are subjected to



different temperature and humidity levels than the interiors. Depending on the depth and intensity of heat and moisture sources, wall surfaces experience differential contraction and expansion.

The density and rigidity of materials used to construct a wall affect the expansion and contraction of each material. Layered surface coatings work as a system. The rigid substrate expands and contracts at a different rate than the upper layers. When the desired aesthetic effect is a smooth layer of surface finish, smaller silt and clay sized particles are required to achieve this effect. The underlying layer of plaster is formulated to contain a greater quantity of slightly larger sand particles. The larger quantity of sand sized particles makes this intermediate layer serve as a buffer between the aesthetically pleasing finish coat and the more rigid masonry substrate.

In the short term, the engineering of Ancestral Puebloan plaster formulations contributed to the durability of these surface finishes at Mesa Verde. Yet it is not a perfect system. The engineering that has contributed to 800 years of durability is also what determines the current method of deterioration for these finishes. In order to achieve the smooth, refined aesthetic found in finish layers at Mesa Verde, a high percentage of silt and clay sized particles are used. Due to the size of these particles, the moisture activated expansion/contraction coefficient is high. The intermediate layer consists of more sand, which acts in the same manner as it would in a ceramic vessel. In ceramics, sand is added to the clay mixture as temper to reduce the shrinkage of pottery as it dries. In the formulation of surface finishes at Mesa Verde, the high sand content of mortars and plasters makes this tripartite system complete. The masonry provides a rigid substrate





that experiences little expansion and contraction. The mortar and subsequent plaster layer are formulated with higher amounts of silt and sand. Thus, they expand and contract less than the masonry. The finish layers of wash are made with a high quantity of smaller particles, making them expand and contract more than underlying plaster layers.

In this way, the initial finish layers are formulated to shrink less than their overlaying counterparts. Stratified finish layer formulations beginning with a rigid substrate and ending with a wash that has a greater range of expansion and contraction, allows intermediate plaster layers whose range of movements is midway between that of the substrate and the wash to serve as a buffer between the two. As such the formulation of this plaster layer restrains the movement of the thinner wash layer. This restraint limits the amount of shrinkage cracks that occur during initial application of the wash layer, but also causes strain within the wash layer. The stress induced by restrained thermal and moisture coefficients of expansion eventually strain the finish layers to the point where they suffer deterioration from these forces. This deterioration occurs at the interfacial bond between layers when one layer moves faster than the other causing shear stresses at the interface between layers.

When the cohesive forces of attraction between particles within a finish layer are stronger than the adhesive forces from one layer to the next and this attraction holds the particle in a rigid structure, environmental stresses cause interfacial cracking. If the same forces were acted upon a flexible structure, the particles would be able to slide over one another instead of cracking. In the case of the clay binders used at Mesa Verde, water serves as a plasticizer between the clay particles making them more flexible. Yet, when



the water evaporates, the structure becomes more rigid and slight environmental changes now induce enough stress to crack this rigid structure.

### **3.2 Mesa Verde Ancestral Puebloan Surface Finishes**

Surface finish analysis at Mug House and at Cliff Palace show consistencies in the formulation of particle-size ratios. The same principle of rigid primer and flexible overlayer applies. At Mesa Verde, the primary binder is the clay sized particles and microcrystalline calcite. When these finishes were originally applied, they may have had additional organic binding media. Yet the predominant binding agent visible today is the clay fraction. According to ethnographic observations<sup>4</sup>, traditional Hopi builders applied surface finishes by smoothing them onto the wall with pieces of sheepskin. By wiping a hand or sheepskin over the wall, a smooth surface is formed as the plate-like particles of clay are aligned parallel to the surface of the wall. The more closely packed the clay particles are, the more dense and less permeable the finish layer becomes. In cases where the clay particles are chemically inert, clay particles are held together by electromagnetic forces alone.

To better understand the potential for similar studies at Mesa Verde, two different materials were used to formulate reproduction plasters. In her thesis, Dix notes that two hues of white finish are present at Mug House. She states that “the white band in Kiva C is translucent and bright white, while the white upper field in Room 28 is opaque and has

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<sup>4</sup> Personal communication with Sally Cole, Archeologist surveying the iconography of Rock Art at Mesa Verde, 3/4/2002.



a yellowish cast.”<sup>5</sup> Dix attributes this color difference to the thickness of paint layers. Further analysis conducted by the University of Pennsylvania attributes this color difference to the presence of gypsum in this “bright white” finish and the presence of calcium carbonate for the white finish with a “yellowish cast”. While layer thickness does affect translucence, the experimental archaeology portion of this research produced additional data. Soil samples obtained from within the park were used to reproduce prehistoric plasters. Two samples of soft white stone were ground with mortar and pestle, then sorted according to particle size and combined to formulate a 10:90 sand:silt/clay particle size distribution. They were then wetted and applied to a base coat of reproduced reddish brown plaster formulated with 60:40 sand:silt/clay particle size distribution. The resulting plasters were classified according to Munsell color. The result was reproduction plasters that simulate the description noted by Dix.

Based on limited data generated by the Slater<sup>6</sup> and Dix<sup>7</sup> surveys, it is difficult to ascertain whether particle-size distribution was consciously modified to produce more durable surface treatments. With further analysis, it would be interesting to explore whether particle size in plaster formulation is simply a product of access to materials as has been suggested by Mary Griffiths or if it is the product of technological innovations. The sourcing of materials for mortar samples led Griffiths to river drainages as a source of materials because this was where the mixture of materials was found. This leads us to theorize that either the source of materials was drainages where erosion had deposited

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<sup>5</sup> Dix, Linnaea A. “Characterization and analysis of Prehistoric earthen plasters, mortars, and paints from Mug House, Mesa Verde National Park Colorado” 1996, p. 95.

<sup>6</sup> Slater, Mary E. “Characterization of earthen architectural surface finishes from Kiva Q, Cliff Palace, Mesa Verde National Park Colorado”, University of Pennsylvania Masters Thesis, 1999.



many different types of soils or that the soils were gathered from their formational sources and manually mixed to form the mortar mixtures used at Mesa Verde. Durability and aesthetic concerns affect the formulation of modern surface treatments, yet with modern methods of paint production and distribution many amateur painters are not familiar with the formulation of the paints that they apply. Prehistoric people did not have the luxury of modern methods for mass production. Thus, it makes sense that prehistoric paint formulation held an empirical basis that was passed on from generation to generation.

Traditional surface finishes have utilized a number of different binding mediums. Plant, mineral and animal based binders have been used to produce surface coating since ancient times. Different names were given to specific paint formulations and these names are not always consistent from one author to the next. Many combinations of mineral, plant and animal proteins were used as surface finishes and to create architectural reliefs. Because these materials are film forming when used in certain combinations, they have been used in paintings, photography and wall finishes. Many of these materials are activated by exposure to temperature and moisture changes. Adhesive binders of animal or plant origin generally have a polymeric structure. These polymers form films by either condensation polymerization or addition polymerization. However, the crosslinking of these natural polymers occurs by addition polymerization.<sup>8</sup> An abundance of traditional formulations uses these polymers as a binding medium combined with either clay or

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<sup>7</sup> Dix, Linnaea A. "Characterization and analysis of Prehistoric earthen plasters, mortars, and paints from Mug House, Mesa Verde National Park Colorado" 1996.

<sup>8</sup> Wilks, Helen et al. *Science for Conservators: Adhesives and Coatings*, Volume 3, Conservation Unit of the Museums & Galleries Commission and Routledge, 1992.





calcium carbonate for the functional use of pigment, temper, filler and bulking agent.<sup>9</sup> Further discussion of the binding agents used to formulate surface finishes is provided in chapter 9 of this study.

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<sup>9</sup> Thornton, Jonathan. "A Brief History and Review of the Early Practice and Materials of Gap-Filling in the West", *Journal of American Institute for Conservation* 1998, Volume 37, Number 1, Article 2, p. 3-22.



## Chapter 4

### PRIOR SURFACE FINISHES RESEARCH AT MESA VERDE

Prior surface finish studies were cited as background research for the formulation of reproduction plasters. At the macroscopic level, separation of one layer from the next is difficult and often results in the contamination of one layer by the next. However, microscopic measurements have determined a different particle size distribution within mortars and layers of earthen surface finish. The University of Pennsylvania defines plasters as finishes measuring more than 1 mm. Thick. The historic term wash is used for layers of finish that measure less than 1 mm. thick. Although further research on this subject must be obtained to support this conclusion, a survey of current literature on this subject suggests that particle size is also related to film thickness. Though not easily assessed in the field, from a technological perspective, particle size may be a better way to define the difference between plaster and wash. Yet, the technological and theoretical use of materials by different social groups may have changed from site to site.

A survey of previous mortar and surface finish analysis was conducted and confirmed by the author. This survey included analysis of samples of prehistoric Mesa Verde mortars, plasters and washes obtained from Mug House, Spruce Tree House, Cliff Palace and Square Tower House. The results of this survey showed a minor variation between the particle size of mortars and plasters, and showed a large difference between the sand:silt/clay ratio for wash layers from the ratio for plaster layers. The mathematical mean of particle size recorded in these studies and the combined mode for both studies are listed in table 1.



**TABLE 1**  
**Sand:Silt/Clay Ratios for Earthen Surface Finishes at Mesa Verde**

Study	Dix, 1996	Slater, 1999	Mode Ratio
Site	Mug House Kiva C	Cliff Palace Kiva Q	Combined Studies
Plaster	54:46	52:48	60:40
Wash	32: 68	16:94	10:90

Slater measured the particle size distribution of plaster layers, defined by layer thickness, from the analysis of five samples.<sup>10</sup> The particle size mathematical mean for these plasters is 52% sand to 48% silt and clay size particles. With the exception of a tan colored plaster only found in sample 11, all plasters were of a buff color. All other particle size distributions for wash layers measured less than or equal to 5% sand sized particles. It is notable that this buff plaster is the most common color used in Cliff Palace. It may be common for premixed quantities of this plaster to be on hand while additional structures were being constructed.

The mean values for particle size distributions in Kiva C of Mug House is based on four samples containing data for a total of 11 plaster layers and 27 wash layers.<sup>11</sup> Similar to an anomaly found in the Slater data regarding buff colored plaster, Dix recorded higher variation in the granulometry in Mug House Kiva C as well as in thin layers of "Orange/Brown" surface treatments using the same formulations for wash layers as were used for plaster layers. Taking this into consideration, the ratio of sand to

<sup>10</sup> Slater, Mary E. "Characterization of earthen architectural surface finishes from Kiva Q, Cliff Palace, Mesa Verde National Park Colorado", University of Pennsylvania Masters Thesis, 1999, p. 102-106.

<sup>11</sup> Dix, Linnaea A. "Characterization and analysis of Prehistoric earthen plasters, mortars, and paints from Mug House, Mesa Verde National Park Colorado" 1996, p. 142-147.



silt and clay particles would drop to 22% sand and 88% silt and clay. Additionally, Dix analyzed an “Orange/Brown” colored mortar sample from Room 28 at Mug House and a soil sample obtained from the Adobe Cave. Particle size distribution for the mortar sample was 60% sand and 40% silt and clay sized particles. The soil sample exhibited a distribution of 95% sand with 5% silt and clay.<sup>12</sup>

It was determined at the outset of this project, that limited materials data was needed to confirm the findings of prior surveys. Since prior studies are the basis for this research, only a bulk gravimetric characterization of plaster samples was obtained with samples from samples obtained at Square Tower House. Preservation of original fabric was deemed more important than specific data acquired through triangulated point proveniencing of surface finish samples. Therefore, samples were obtained non-destructively by collecting spalls that had already naturally detached from their substrate. Consequently, provenience information is limited to the room from which they were collected. This collection strategy does not provide the level of detailed provenience information needed for full surface finish analysis. Since full surface finish analysis is not the purpose of this study, this method of sample collection provided suitable data for a comparison to bulk gravimetric analysis noted by Dix. A comparison of bulk gravimetric particle size analysis is provided in table 2. Additional characterization of original plasters is interspersed as a reference for comparison to the stabilization soils used in this study.

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<sup>12</sup> Dix, Linnaea A. “Characterization and analysis of Prehistoric earthen plasters, mortars, and paints from Mug House, Mesa Verde National Park Colorado” 1996, p. 148.





TABLE 2

Comparison of Particle Size Distribution for Reddish Brown Plaster Samples

Sieve Number	Diameter	Square Tower House 5MV650 Kiva D (Carr, 2002)	Mug House Kiva 5MV1229C Plaster (Dix, 1996)	Mug House Room 5MV122928 Mortar (Dix, 1996)
8	2.36 mm	0%	0%	3%
16	1.18 mm	0%	0%	0%
30	600 Mm	0%	0%	0%
50	300 Mm	1%	0%	0%
100	150 Mm	10%	9%	9.00%
200	75 Mm	43%	40%	28%
Fines / Pan	NA	46%	50%	57%

The combined values for surface finish analysis from both the Dix and Slater studies yielded a modal average of 60% fine sand with 40% silt and clay particle size for earthen plasters and 10% fine sand combined with 90% silt and clay particle size for earthen washes. A variety of colors were recorded and both studies noted the presence of calcium in their samples.<sup>13</sup> This is not surprising since the use of calcium carbonate (lime) and calcium sulfate (gypsum) have been used in the manufacture of mortars and surface finishes from prehistoric times to modern day construction. Furthermore, of the four surface finish samples subjected to x-ray diffraction in the Getty study three of the

<sup>13</sup> Slater, Mary E. "Characterization of earthen architectural surface finishes from Kiva Q, Cliff Palace, Mesa Verde National Park Colorado", University of Pennsylvania Masters Thesis, 1999, p. 49. & Dix, Linnaea A. "Characterization and analysis of Prehistoric earthen plasters, mortars, and paints from Mug House, Mesa Verde National Park Colorado" 1996, p. 95.



samples indicated the presence of calcium and no gypsum, while testing of the fourth sample revealed the presence of gypsum with only trace evidence of calcium.<sup>14</sup>

One color that was consistently formulated in both the study of Mug House Kiva C and Cliff Palace Kiva Q was the white wash. When studying the materials in Ancestral Puebloan white surface finishes, Watson Smith identified a binder of kaolin and/or other silicacious soils mixed with a ground pigment of either gypsum or calcium carbonate.<sup>15</sup> Samples of white kaolinite, calcium carbonate, and a white sandstone from the Menefee formation were obtained at Mesa Verde for this study.<sup>16</sup> (see table 3) The sample of calcium carbonate deposit (see figure 1) was selected to reproduce the white wash used in this study. For comparative purposes, the sandstone sample was also subjected to materials testing.

Every layer of white wash in the Mug House and Cliff Palace studies were formulated with 5-10% sand and 90-95% silt and clay sized particles. The only variation in particle size between these studies indicated that the Mug House Kiva C white washes consistently contained 5% more sand particles than the white washes used at Cliff Palace.

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<sup>14</sup> Mueller, Urs. "Final Report", The Getty Conservation Institute. Los Angeles, CA, 11/29/2001.

<sup>15</sup> Smith, Watson. "Kiva Mural Decorations at Awatovi and Kawaika-A", Appendix A: Analyses of the Component Materials of Pueblo Mural Paintings, Peabody Museum, Cambridge, MA, 1952, p.34.

<sup>16</sup> Roch identifications made by personal communication with Mary Griffiths, Geologist, 5/16/2002.



TABLE 3

**Mesa Verde Soil Samples used to Formulate White Surface Finishes**

Sample Name	MR-1	SY-1
Description	White deposit of kaolinite	Caliche deposit on yellow sandstone
Sampling Location	Mile Marker 12.5 of Main Park Road	Mesa Loop, Balcony House
Material Color	7.5YR8/1 White	10YR8/2 White
Soluble Salt Content	Not Tested	70% Carbonate content
Resulting Plaster Color	7.5YR8/1 White	2.5YR8/2 Pale Yellow

**4.1 Materials Characterization of Soils and Surface Finishes**

Petrographic analysis of materials used to formulate Mesa Verde's original earthen mortars was conducted by geologist Mary Griffiths. She concluded that the soils used for both mortars and plasters at Mesa Verde were likely obtained from local sources within the park.<sup>17</sup> Local soils have been used by park staff in the periodic stabilization of structures since 1906.<sup>18</sup> Four different colored stabilization soils are currently used at Mesa Verde. These materials are not consistently obtained from the same location,<sup>19</sup> so the particle size distribution and other soil characteristics may vary from one delivery to the next. The primary soils used in this study were obtained from the stabilization crews' supply. The four stabilization soils used at Mesa Verde during this study are (1) a red soil collected from Weatherill Mesa, (2) a yellow soil obtained locally from a quarry on the adjoining Ute Reservation, (3) white caliche obtained from road cuts in the vicinity of

<sup>17</sup> Nordby, Larry and Mary Griffiths. "Cliff Palace Building Materials Classification and Selection", December, 1999.

<sup>18</sup> Personal communication with Kathy Fiero, stabilization archeologist at Mesa Verde, 3/13/2002.



Balcony House and (4) commercially obtained quartz sand. Armed with this information, the soils were collected and characterized according to particle size distribution, acid solubility, salt content and Munsell color<sup>20</sup>.

#### **4.2 Mortar Analysis**

Positive results for a presence of calcium carbonates justified further testing to quantify the amount of acid soluble content contained within native soils versus the amount contained within original surface finishes. Wet chemical gravimetric mortar analysis was conducted according to the procedure outlined in Experiment #21: Mortar Analysis: Simple Method as specified in Teutonico (1988).<sup>21</sup>



Figure 1. Mortar analysis and substrate porosity testing at the University of Pennsylvania

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<sup>19</sup> Personal communication with Kathy Fiero, stabilization archeologist at Mesa Verde, 3/13/2002.

<sup>20</sup> American Society for Testing and Materials. "ASTM D1535-01 Standard Practice for Specifying Color by the Munsell System", 2001. <http://www.astm.org/cgi-bin/SoftCart.exe/index.shtml?E+mystore>

<sup>21</sup> Teutonico, Jean Marie. "ARC: A Laboratory Manual for Architectural Conservators", ICCROM, 1988, p. 113-116.





A single sample of each soil currently used by Mesa Verde's stabilization crew was tested. Former work conducted by the University of Pennsylvania has documented a distinct difference between the sand:silt/clay ratio used to produce a plaster from the sand:silt/clay ratio used to manufacture a wash at Mesa Verde. Thus, additional tests were conducted (see figure 1). A sample of prehistoric plaster that had spalled off the wall of Kiva C in Square Tower House was used for this test.<sup>22</sup> Tests were also run with silt fraction samples of both the yellow/very pale brown and red/reddish brown soils to approximate how much of the acid soluble material is contained within the silt fraction.

A sample of each soil was weighed, then wetted with de-ionized water and hydrochloric acid. Observations were recorded as the acid-soluble fraction chemically reacted to form a gas. Once the reaction was complete, the fine portion and coarse portion of each sample were weighed to gravimetrically determine the percentage of acid soluble materials, the percentage of fine particles (see figure 2) and percentage of coarse particles (see figure 3). Mortar analysis test methods dictate that the coarse particles be further classified according to particle size. Since particle size distribution of non-chemically treated Reddish Brown soil was already determined according to ASTM D 422-63(1998) Standard Test Method for Particle-Size Analysis of Soils, these results are displayed side by side for easy comparison (see tables 4, 5, and 6).

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<sup>22</sup> Provenience attributed according to Mesa Verde Save America's Treasures Map: Brisben & Burnett 2001.



**TABLE 4****Particle Size Analysis for Untreated and Acid Treated Reddish Brown Samples**

Sieve size		Untreated Reddish Brown Soil Sample		Treated Reddish Brown Soil Sample	
Sieve No.	Diameter	Weight Retained	% Retained	Weight Retained	% Retained
8	2.36 mm	0.00 g	0%	0.00 g	0%
16	1.18 mm	0.00 g	0%	0.00 g	0%
30	600 Mm	0.00 g	0%	0.01 g	0%
50	300 Mm	3.44 g	1%	0.12 g	1%
100	150 Mm	59.61 g	16%	1.90 g	10%
200	75 Mm	67.46 g	18%	3.04 g	15%
Pan & Fines	NA	297.35	64%	13.35 g	68%
Acid Soluble	NA	NA	NA	NA	5%

**TABLE 5****Particle Size Analysis for Untreated and Acid Treated Light Brown Samples**

Sieve size		Untreated Light Brown Plaster Sample		Treated Light Brown Plaster Sample	
Sieve No.	Diameter	Weight Retained	% Retained	Weight Retained	% Retained
8	2.36 mm	0.00 g	0%	0.00 g	0%
16	1.18 mm	0.00 g	0%	0.00 g	0%
30	600 Mm	0.00 g	0%	0.10 g	1%
50	300 Mm	1.47 g	1%	0.14 g	1%
100	150 Mm	15.32 g	10%	1.11 g	6%
200	75 Mm	65.71 g	43%	5.20 g	26%
Pan & Fines	NA	70.30 g	46%	11.63 g	58%
Acid Soluble	NA	NA	NA	NA	8%



TABLE 6

## Particle Size Analysis for Acid Treated White and Light Brown Soil Samples

Sieve size		Treated White (SY-1) Soil Sample		Treated Very Pale Brown Soil Sample	
Sieve Number	Diameter	Weight Retained	% Retained	Weight Retained	% Retained
8	2.36 mm	0.40 g	2%	0.00 g	0%
16	1.18 mm	0.12	1%	3.10 g	16%
30	600 Mm	0.01	0%	2.81 g	14%
50	300 Mm	0.01	0%	2.10	11%
100	150 Mm	0.02	0%	2.64	13%
200	75 Mm	0.03	0%	2.20	11%
Pan & Fines	NA	5.36	27%	4.60	3%
Acid Soluble	NA	NA	70%	NA	32%



Figure 2. Silt and clay fraction of the  
Reddish Brown Soil

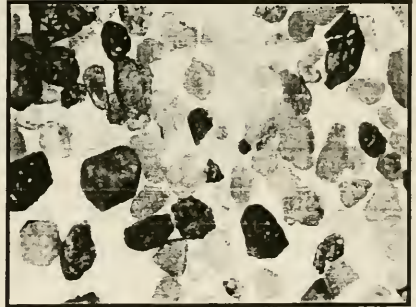


Figure 3. Fine grained sand particles  
magnified 500X

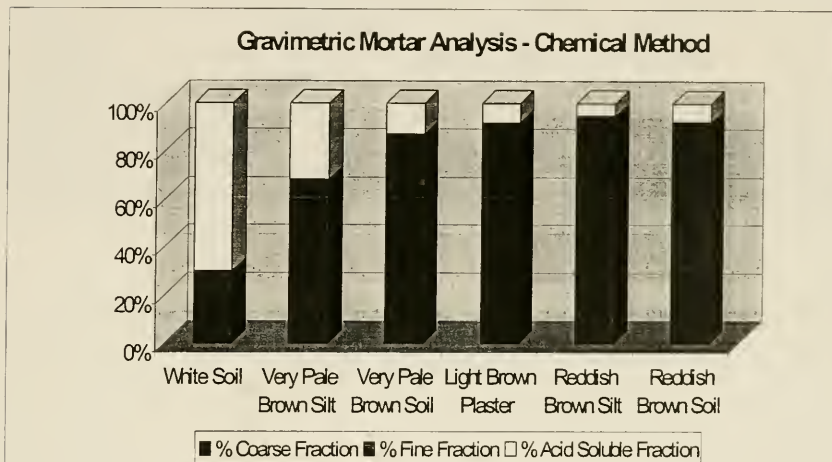
Mortar testing confirmed that the white caliche contained more acid soluble material in its silt/clay fraction. Nearly 70% of the white caliche sample (SY-1) consists of acid soluble material. A majority of this acid soluble material is presumed to be the calcium carbonate identified in chemical spot testing conducted for this study. The quantity of acid soluble material in the white sample is in stark contrast to the 5% acid soluble content within the reddish brown soil sample while the acid soluble content of very pale brown soil was identified as an intermediary 32% (see table 7). Based these results, two soils were selected to reproduce



earthen surface finishes. The white sample was selected to reproduce earthen finishes similar to those identified by Dix as having a microcrystalline calcite binder.<sup>23</sup> The reddish brown soil was selected to reproduce earthen finishes identified by Smith as having a silicacious binder.<sup>24</sup>

**TABLE 7**

**Comparison of Results for Mortar Analysis by Wet Chemical Method**



<sup>23</sup> Dix, Linnaea A. "Characterization and analysis of Prehistoric earthen plasters, mortars, and paints from Mug House, Mesa Verde National Park Colorado" 1996.

<sup>24</sup> Smith, Watson. "Kiva Mural Decorations at Awatovi and Kawaika-A", Appendix A: Analyses of the Component Materials of Pueblo Mural Paintings, Peabody Museum, Cambridge, MA, 1952.





**TABLE 8****Acid Soluble Fraction Determined by Mortar Analysis by Wet Chemical Method**

<b>Mortar Analysis: Wet Chemical Method</b>	<b>Original weight</b>	<b>Reaction: Visual Observations</b>	<b>Fine fraction weight</b>	<b>% Fine fraction Weight</b>	<b>Coarse fraction weight</b>	<b>% Coarse fraction weight</b>	<b>% Acid Soluble Weight</b>
White Soil (SY-1)	20.00 g	Very strong, long term	5.36	26.80%	0.59 g	2.95%	70%
Very Pale Brown Silt (SY-2)	20.00 g	Very strong, long term	13.68 g	68.40%	0 g	0%	32%
Very Pale Brown Soil (SY-2)	20.00 g	Very strong, long term	4.3 g	21.50%	13.15 g	65.75%	12.75%
Light Brown Plaster (SQT-10)	20.00 g	Strong, long term	10.99 g	54.95%	7.40 g	37%	8%
Reddish Brown Silt (SY-3)	20.00 g	Clearly, but not long term	18.93 g	94.65%	0 g	0%	5%
Reddish Brown Soil (SY-3)	20.00 g	Clearly, but not long term	12.35 g	61.75%	6.07 g	30.35%	8%



## Chapter 5

### PARTICLE SIZE ANALYSIS OF STABILIZATION SOILS

Particle size analysis of the soils used in this study were conducted to establish a basis for reproducing the plaster formulations documented in surface finish analysis. Particle size was determined according to ASTM D422-63(1998) Standard Test Method for Particle-Size Analysis of Soils. Further basis for this procedure is contained within Experiment #18A: Particle Size Analysis: Part I / Sieving Procedure as specified in ARC: A Laboratory Manual for Architectural Conservators.<sup>25</sup> A Combustion Engineering Model RX-86 Sieve Shaker was used to sort each sample by particle size.

The red and yellow soils used by the Mesa Verde stabilization crew are obtained in loose form while the white caliche (calcium carbonate) is collected as a weakly bound stone and then ground to produce soil of the desired particle size. Therefore, a sample of the Reddish Brown soil was characterized according to particle size distribution (see tables 9, 10, and 11), with only a sample of the yellow/Very Pale Brown soil being characterized as a basis for comparison (see figures 12, 13, and 14). Petrographic analysis confirmed that the commercially graded stabilization sand is predominantly composed of well sorted quartz grains (see figure 4). This sand was not tested since it is used as an aggregate additive in mortar formulations and has no bearing on this study.

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<sup>25</sup> Teutonico, Jean Marie. "ARC: A Laboratory Manual for Architectural Conservators", ICCROM, 1988, p. 73.



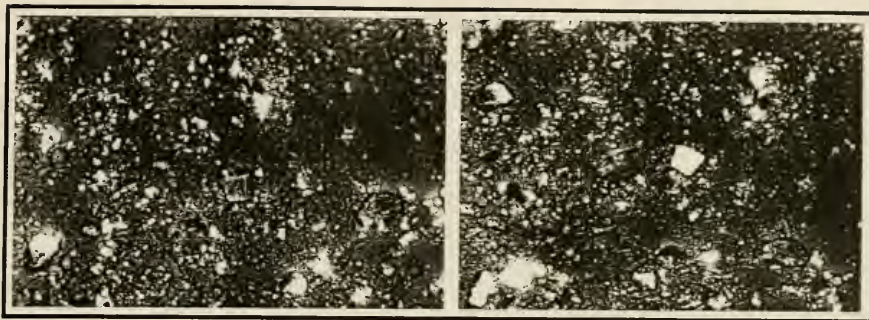


Figure 4. Photomicrographs of fine quartz sand particles displaying birefringence

Particle size analysis revealed that the very pale brown soil was substantially better graded than that of the reddish brown soil. Since both soils were obtained from the Mesa Verde stabilization crew mortar and repointing supplies, the reddish brown soil may already have been sieved. It was immediately apparent that these soils were not properly graded for the reproduction of earthen surface finishes and would have to be modified in order to produce accurate facsimiles (see tables 9, 10, 11 and 12).

### **5.2 Pre-Sieved Reddish Brown Stabilization Soil Sample (SY-3)**

Total bulk weight before sieving = 370.00 g.

Weight of pre-sieved portion of soil sample = 193.86 g.

Total bulk weight after sieving = 366.89 g

Weight of post-sieved portion of soil sample = 192.48 g.

Sand (diameter 2.36 mm. - 75 Mm.) = 130.51 g.

Weight of suspended fines = 173.41 g.

Silt/Clay (diameter < 75 Mm.) = 235.38 g.

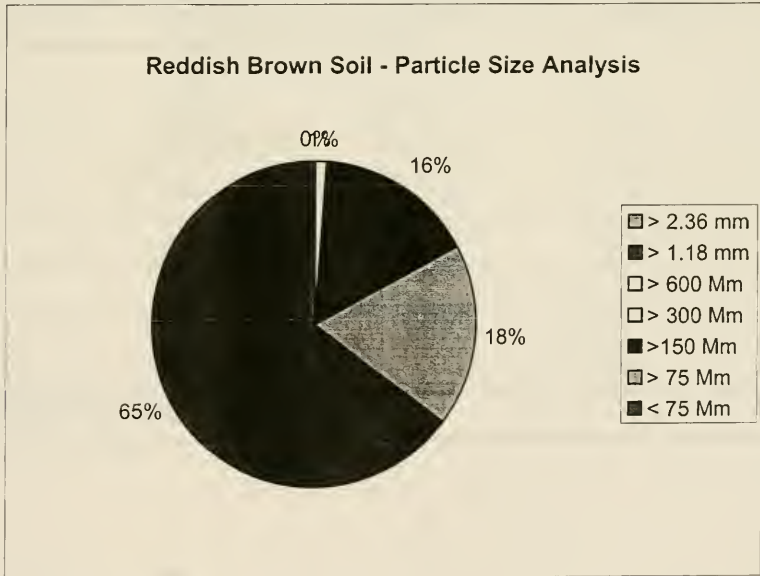
Munsell Color = 5YR5/4 Reddish Brown



**TABLE 9**  
**Particle Size Analysis for Reddish Brown Soil Sample (SY-4)**

Sieve Number	Diameter	Weight Retained	% Retained
8	2.36 mm	0.0 g	0%
16	1.18 mm	0.0 g	0%
30	600 Mm	0.0 g	0%
50	300 Mm	3.44 g	1%
100	150 Mm	59.61 g	16%
200	75 Mm	67.46 g	18%
Pan	NA	61.97 g	17%
Fines	Suspension	174.41 g	47%
<b>Sand to Silt/Clay Ratio = 36:64</b>			

**TABLE 10**  
**Graphic Distribution of Particle Size Analysis - Reddish Brown Soil Sample**







### 5.1 Yellow/Very Pale Brown Stabilization Soil Sample (SY-2)

Total bulk weight before sieving = 400.00 g.

Weight of pre-sieved portion of soil sample = 265.6 g.

Total bulk weight after sieving = 398.02 g.

Weight of post-sieved portion of soil sample = 263.62 g.

Sand (diameter 2.36 mm. - 75 Mm.) = 256.72 g.

Weight of suspended fines = 134.4 g.

Silt/Clay (diameter < 75 Mm.) = 141.3 g.

Munsell Color = 10YR7/4 Very Pale Brown

**TABLE 11**

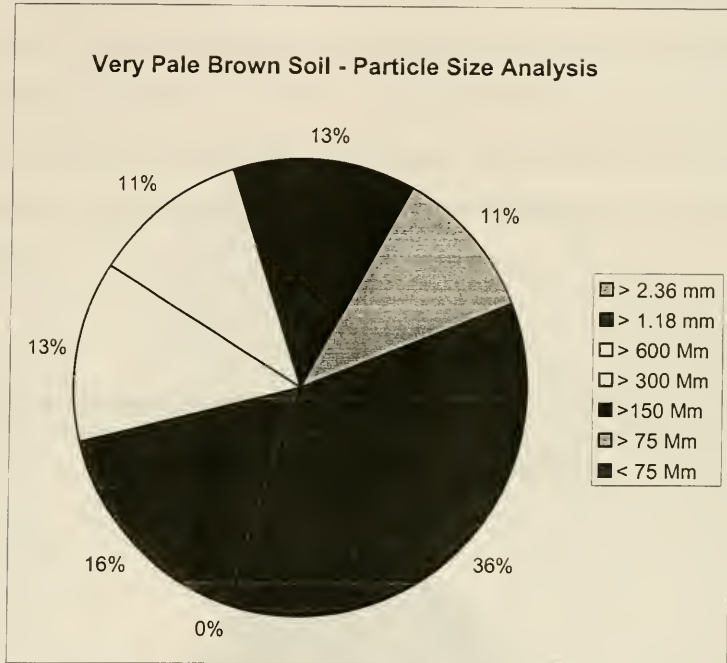
#### **Particle Size Analysis for Yellow/Very Pale Brown Soil Sample (SY-2)**

Sieve Number	Diameter	Weight Retained	% Retained
8	2.36 mm	1.51 g	0%
16	1.18 mm	62.21 g	16%
30	600 Mm	53.59 g	13%
50	300 Mm	45.21 g	11%
100	150 Mm	50.78 g	13%
200	75 Mm	43.42 g	11%
Pan	NA	6.9 g	2%
Fines	Suspension	134.4 g	34%
Sand to Silt/Clay Ratio = 64:36			



TABLE 12

## Graphic Distribution of Particle Size Analysis - Very Pale Brown Soil Sample



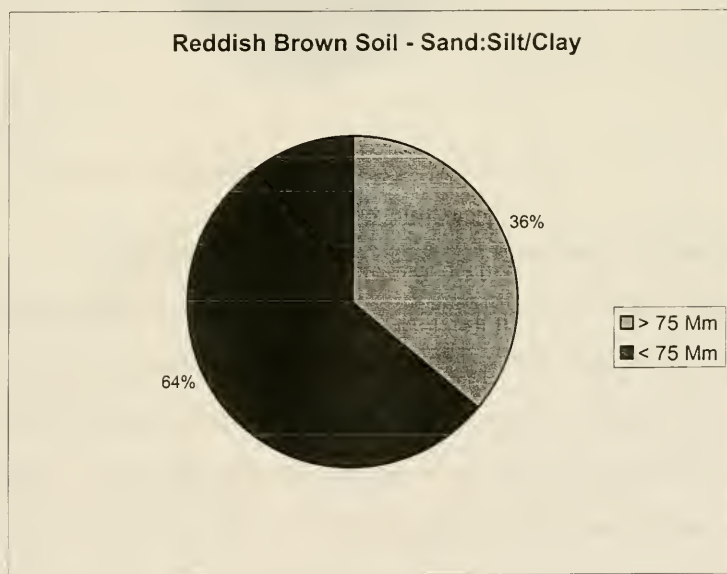
Based on information compiled from previous surface finish analysis studies, the high silt and clay content of this soil was not appropriate for unammended use as facsimile earthen plaster. Use of this soil without adding fine grained sand would result in shrinkage cracking and poor surface finish durability. This assertion was confirmed by the results of two accelerated weathering experiments. The accelerated weathering experiment described in chapter seven utilized soil formulations that were not based upon the results of prior surface finish analysis. These samples exhibited severe deterioration in only three weathering cycles. While the accelerated weathering experiment described



in chapter eight produced less deterioration after being subjected to four cycles of increasingly severe environmental fluctuations. Thus, it is safe to conclude that the Ancestral Puebloan surface finish formulations set forth in this study were intentionally formulated for durability. This assertion is also supported by the crushed aggregate particles identified microscopically.<sup>26</sup> The presence of crushed aggregate particles further indicates that Ancestral Puebloan earthen surface finish formulations were intentionally modified.

**TABLE 13**

**Graphic Distribution of Sand:Silt/Clay Ratio- Reddish Brown Soil Sample**

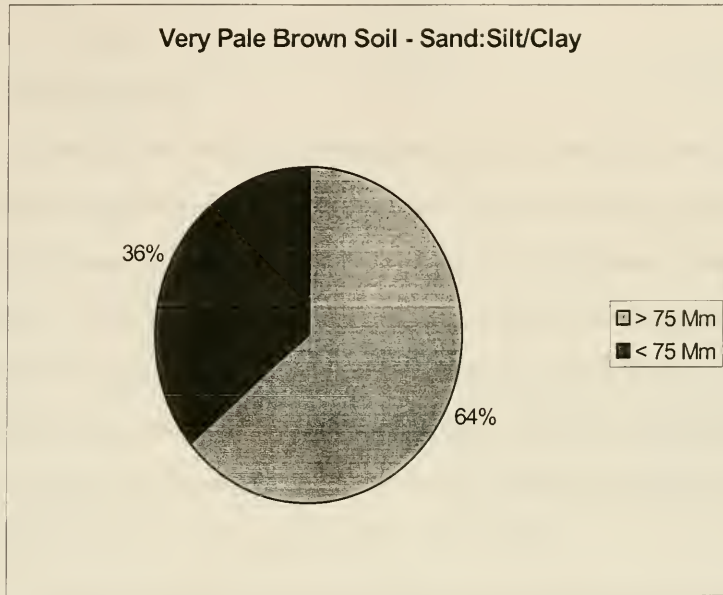


<sup>26</sup> Slater, Mary E. "Characterization of earthen architectural surface finishes from Kiva Q, Cliff Palace,



TABLE 14

## Graphic Distribution of Sand:Silt/Clay Ratio - Very Pale Brown Soil Sample



Particle size distribution, petrographic analysis, mortar analysis, chemical and elemental analysis each contribute valuable information when sourcing and subsequently reproducing the earthen surface finishes at Mesa Verde National Park. A survey of prior finish analysis provided specific formulations for the reproduction of these finishes. Conducting particle size analysis of local soils is the first step in mixing these surface finish formulations. Once the sand:silt/clay ratio of each soil was obtained, the sand fraction was separated from the silt fraction according to the procedure set forth in ASTM D422-63(1998). The silt/clay fraction of reddish brown soil measuring less





than 75 Mm in diameter, caliche deposits ground to the same sized silt/caly fraction and a fine quartz sand that measured between 75 Mm and 150 Mm were selected to mix the reproduction finish formulations set forth in this study.

### **5.3 Test for Salts**

Six samples were tested for salts with Merckoquant © test strips. Each sample was prepared with one gram of sample combined with 2 cc. de-ionized water. The sample was mixed thoroughly then allowed to settle for 15 minutes before being subjected to the following tests: chlorid test strip #1.10079.001, nitrate test strip #1.10020.001, sulfate test strip 1.10019.000 and nitrate test strip 1.10020.000. After determining positive results for Nitrates, this experiment was followed with chemical tests as outlined in the procedures for Experiment #16: Qualitative Analysis of Water-Soluble Salts and Carbonates as specified in Teutonico (1988).<sup>27</sup>

The procedure was to place each of the test strips into the solution and allow them to dry. A reading was taken at 5 minutes to determine if chemical confirmation was required. The most common reaction after 5 minutes was a positive test for Nitrates. Thus, all samples were subjected to a chemical test for Nitrates. (Readings for strips must be calculated for grams per liter.) The chemical test for  $\text{NO}_2^-$  was to add two drops of dilute acetic acid ( $\text{CH}_3\text{COOH}$  2N) and two drops of Griess-Hosvay's Reagent. An intense pink color would indicate a positive reaction. Addition of zinc powder to the same solutions reduced  $\text{NO}_3^-$  to  $\text{NO}_2^-$  thus producing the same positive reaction. Results for the above mentioned tests are provided in table 15.

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<sup>27</sup> Teutonico, Jean Marie. "ARC: A Laboratory Manual for Architectural Conservators", ICCROM, 1988, p. 58.



**TABLE 15**  
**Qualitative Analysis of Salts**

Sample:	EM Quant $\text{NO}_2^-$	EM Quant $\text{NO}_2^-/\text{NO}_3^-$	EM Quant $\text{SO}_4^{2-}$	EM Quant $\text{Cl}^-$	Chemical $\text{NO}_2^-$	Chemical $\text{NO}_3^-$
Stabilization Sand	0 g/l	-/0 mg/l	< 200 (-)	0	-	-
White Sandstone Sand	0 g/l	-/0 mg/l	< 200 (-)	0	-	-
Reddish Brown Soil Sand	0 g/l	-/0 mg/l	< 200 (-)	0	-	-
Square Tower Plaster	0 g/l	++/25 mg/l	< 200 (-)	500 mg/l	-	-
Square Tower Sandstone	0 g/l	-/0 mg/l	< 200 (-)	0	-	-
Caliche	0 g/l	-/0 mg/l	< 200 (-)	0	-	-
White Sandstone Binder	0.1 g/l	-/0 mg/l	< 200 (-)	0	-	+-
Reddish Brown Soil Silt	0 g/l	-/0 mg/l	< 200 (-)	0	-	-
Weathered Red Plaster/Wash	0 g/l	+/10 mg/l	>400 (+)	500 mg/l	-	-
Brick Substrate	0 g/l	-/0 mg/l	< 200 (-)	500 mg/l	-	-

**KEY:**

+- = concentration of the ion at the limit of perceptibility

- = absence of the ion

+ = presence of the ion



The presence of salts at Mesa Verde National Park contributes to the deterioration of earthen surface finishes. These salts are present in both the soils that are used to formulate original surface finishes and contained within the stones upon which these finishes are applied. Nitrates, sulfates and chlorides were found at varying amounts within the soils, stones and Ancestral Puebloan plaster finishes at Mesa Verde. These results are significant because the leaching of salts to the surface of masonry structures and the subsequent crystallization of these salts can rapidly accelerate the delamination and detachment of surface finishes (see figures 5 and 6).

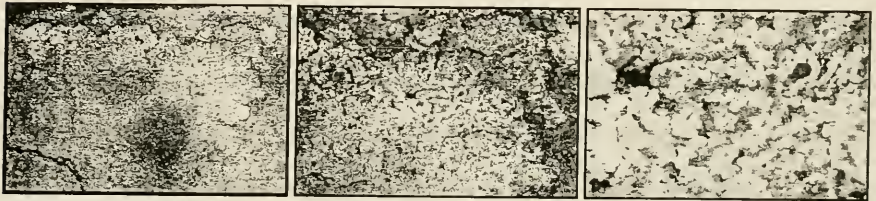


Figure 5. Photomicrographs of salts formed on reproduction earthen finishes applied to a Mesa Verde sandstone substrate, shown at 10x, 20x and 40x magnification.

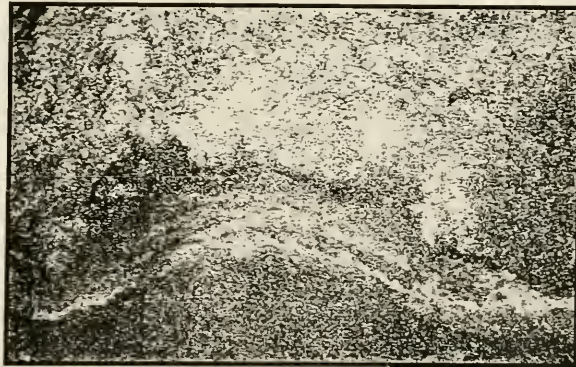


Figure 6. Tide line of salts that have leached out of a sandstone sample obtained from Mesa Verde National Park



## 5.4 Chemical Testing for Calcium Carbonate

Calcium carbonate is a water soluble salt that occurs naturally at Mesa Verde National Park. Similar in chemical composition to gypsum, identification of calcium carbonate in earthen surface finishes contributes to interpretations of the durability and technological application of Ancestral Puebloan surface finishes.

Both calcium carbonate and gypsum have been identified in Ancestral Puebloan surface finish studies.<sup>28</sup> Further studies of this topic may contribute to the modern interpretation of Ancestral Puebloan surface finish application technology.<sup>29</sup> During this study, the author noted that calcium sulfate is considered to be more water soluble than calcium carbonate.<sup>30</sup> An additional solubility test was conducted with caliche samples obtained from Mesa Verde. After three weeks of soaking, a miniscule amount of material separated from the sandstone on which this white substance was deposited. Thus, it is suspected that the presence of gypsum prior to chemical spot testing was minimal.

### 5.4.1 Test for Calcium

A wet chemical spot test was selected that uses nitric acid and sulfuric acid to test for calcium. This was followed up with a test for carbonate using hydrochloric acid and barium hydroxide.<sup>31</sup> The test for calcium uses nitric acid to separate the calcium and

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<sup>28</sup> Smith, Watson. "Kiva Mural Decorations at Awatovi and Kawaika-A", Appendix A: Analyses of the Component Materials of Pueblo Mural Paintings, Peabody Museum, Cambridge, MA, 1952.

<sup>29</sup> Vandiver, Pamela B., James R. Druzik, Jose Luis Galvan Madrid, Ian C. Freestone, George Segal Wheeler. "Materials Issues in Art and Archaeology IV", Materials Research Society: Symposium Proceedings, Volume 352, May 16-21, 1994, Cancun, Mexico, 1995.

<sup>30</sup> Jusko, Don A. "History of Painting Mediums... Glue, Wax Paint, Cera Colla, Mastic, Casein Paint, Fresco, Egg, Oil Paint, Acrylic Paint", <http://www.mauigateway.com/~donjusko/1mediums.htm#100,000 B/C>

<sup>31</sup> Nancy Odegard, Scott Carrol and Werner S. Zimmt, Material Characterization Test for Objects of Art and Archaeology, Archetype Publications Ltd, 2000, p 100-103.





disperse it into a solution. Sulfuric acid combines with calcium ions to form gypsum crystals that are visible with the aid of a microscope (see figure 7).

When calcium ions ( $\text{Ca}^{2+}$ ) are exposed to certain acids ( $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$ ) they undergo the chemical reaction  $\text{Ca}^{2+}(\text{aq}) + \text{SO}_4^{2-}(\text{aq}) \rightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O}(\text{s})$  resulting in the formation of gypsum crystals ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ). Therefore, the chemical spot test used to determine the presence of absence of calcium is based on the formation of gypsum crystals after the sample has been chemically treated (see table 16).

**Table 16**  
**Chemical Spot Test Results for the Presence of Calcium**

Sample Name	Munsell Color	Test Result
White Soil (SY-1.1)	10YR8/2	Positive
White Soil (SY-1.2)	10YR8/2	Positive
White Soil (SY-1.3)	10YR8/2	Positive
White Sandstone (MR-1.1)	10YR8/8	Negative
White Sandstone (MR-1.2)	10YR8/8	Negative
White Sandstone (MR-1.3)	10YR8/8	Negative
Reddish Brown Soil (SY-3.1)	5YR5/4	Positive
Reddish Brown Soil (SY-3.2)	5YR5/4	Positive
Reddish Brown Soil (SY-3.3)	5YR5/4	Positive
Kiva D Pinkish White Plaster 5MV650 (SQT-10.4w)	7.5YR8/2 overlaying 7.5YR5/4	Positive
Kiva D Pinkish White Plaster 5MV650 (SQT-10.5w)	7.5YR8/2 overlaying 7.5YR5/4	Positive
Kiva D Pinkish White Plaster 5MV650 (SQT-10.6w)	7.5YR8/2 overlaying 7.5YR5/4	Positive
Kiva D Light Brown Plaster 5MV650 (SQT-10.7r)	7.5RY6/4	Positive
Kiva D Light Brown Plaster 5MV650 (SQT-10.8r)	7.5RY6/4	Positive
Kiva D Light Brown Plaster 5MV650 (SQT-10.9r)	7.5RY6/4	Positive



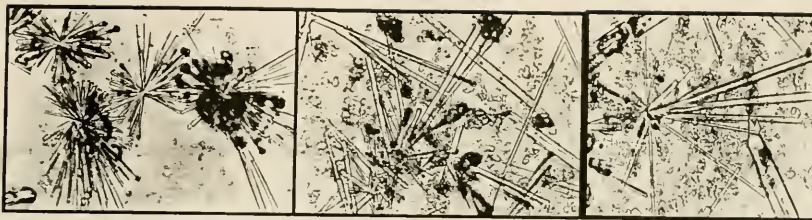


Figure 7. Photomicrographs of gypsum crystals at 40x magnification that confirm a positive chemical reaction for calcium.

#### 5.4.2 Test for carbonates

To test for calcium carbonate and other acid soluble contents, this procedure uses hydrochloric acid and barium hydroxide. Hydrochloric acid (HCl) in contact with carbonate ions ( $\text{CO}_3^{2-}$ ) release carbon dioxide ( $\text{CO}_2$ ) gas as shown in the chemical formula  $\text{CO}_3^{2-}(\text{s}) + 2\text{HCl}(\text{aq}) \rightarrow \text{CO}_2(\text{g}) + \text{H}_2\text{O}(\text{l}) + 2\text{Cl}^-(\text{aq})$ . Barium hydroxide is used to confirm this reaction (see tables 17 and 18).

**TABLE 17**

**Test to Confirm the Presence or Absence of Carbonates in Sample SQT-10 (r)**

Sample Name	Munsell Color	Test Result	Confirmation
Kiva D Light Brown Plaster 5MV650 (SQT-10.1r)	7.5YR5/4	Positive	Positive
Kiva D Light Brown Plaster 5MV650 (SQT-10.2r)	7.5YR5/4	Positive	Positive
Kiva D Light Brown Plaster 5MV650 (SQT-10.3r)	7.5YR5/4	Positive	Positive
Kiva D Light Brown Plaster 5MV650 (SQT-10.4r)	7.5YR5/4	Positive	Positive
Kiva D Light Brown Plaster 5MV650 (SQT-10.5r)	7.5YR5/4	Positive	Positive
Kiva D Light Brown Plaster 5MV650 (SQT-10.6r)	7.5YR5/4	Positive	Positive



TABLE 18

Chemical Spot Test to Determine the Presence or Absence of Carbonates

Sample Name	Munsell Color	Test Result	Confirmation
White Soil (SY-1.1)	10YR8/2	Positive	Positive
White Soil (SY-1.2)	10YR8/2	Positive	Positive
White Soil (SY-1.3)	10YR8/2	Positive	Positive
Reddish Brown Soil (SY-3.1)	5YR5/4	Positive	Positive
Reddish Brown Soil (SY-3.2)	5YR5/4	Positive	Positive
Reddish Brown Soil (SY-3.3)	5YR5/4	Positive	Positive
Kiva D White Plaster 5MV650 (SQT-10.1w)	7.5YR8/2 overlaying 7.5YR5/4	Positive	Positive
Kiva D White Plaster 5MV650 (SQT-10.2w)	7.5YR8/2 overlaying 7.5YR5/4	Positive	Positive
Kiva D White Plaster 5MV650 (SQT-10.3w)	7.5YR8/2 overlaying 7.5YR5/4	Positive	Positive
Kiva D Light Brown Plaster 5MV650 (SQT-10.1r)	7.5YR5/4	Positive	Positive
Kiva D Light Brown Plaster 5MV650 (SQT-10.2r)	7.5YR5/4	Negative	NA
Kiva D Light Brown Plaster 5MV650 (SQT-10.3r)	7.5YR5/4	Negative	NA
Room 24 Light Brown Plaster 5MV640 (STH-2.1r)	7.5YR6/4	Negative	NA
Room 24 Light Brown Plaster 5MV640 (STH-2.2r)	7.5YR6/4	Negative	NA
Room 24 Light Brown Plaster 5MV640 (STH-2.3r)	7.5YR6/4	Negative	NA



## Chapter 6

### PREPARATION OF SAMPLES WITH UNIFORM THICKNESS

An initial survey of laboratory testing methods was conducted at the outset of this research. Many materials performance-testing standards are intended as a basis for comparing commercially manufactured products. This survey identified testing methods established by governmental standards for assessing the performance of architectural materials in the United States and in Great Britain, relative tests to assess materials compatibility developed by the coatings industry, tests to assess soil morphology developed by the US Department of Agriculture, testing used in the fields of food science and biochemistry, analysis methods used in the field of fine arts conservation, and testing methods developed specifically for architectural conservation assessment. American Standard Testing Methods (ASTM), a private distributor of standardized testing methods proved to be the most useful source of materials testing methods. This decision is primarily attributed to the general acceptance of ASTM distributed testing methods and accessibility to ASTM publications at the University of Pennsylvania Engineering Library.

When testing surface finishes, it is important that surface finishes are applied at a uniform thickness. The cohesive strength, rigidity and expansion/contraction coefficient of surface finishes is related to the thickness of the surface coating. This is true of both the surface finishes and the adhesive film applied to reattach it. Previous conservation studies have determined that both film thickness and the uniformity of its application affect its performance. It is well documented that cracking and the eventual deterioration





of painted finishes are likely to occur in areas with less cohesive strength.<sup>32</sup> Minute flaws in the thickness of application can induce cracking in the thinner areas. This is why the coatings industry uses leveling agents in their products. Even brush marks produced during paint application produce thin areas of finish that are more prone to cracking.

Determination of finish thickness was established through documentary research. Facsimile surface finishes were composed of a 0.8 mm. thick application of wash overlaying a 5 mm. thick plaster application and a 25 mm. thick substrate. The consistency of finish formulations used for this study required manual leveling and were not conducive to brush, spray or even wire-rod applications. This consistency is due to particle size distribution of the materials used to formulate reproduction finishes. The only control for viscosity and leveling with these plaster and wash formulations was the amount of water used and the amount of soil particles in suspension at the time of application. Additional problems were encountered while cutting masonry substrates to this degree of precision. Attempts were made to apply plaster and wash formulations according to both ASTM Practice D823-95(2001) Standard Practices for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels and ASTM Test Method D4062-99 Standard Test Method for Leveling of Paints by Draw-Down Method.

After some experimentation, ASTM Test Method D823-95 Practice E did prove successful. Most commercial draw-down tools are designed to apply a thin layer of finish to a larger substrate than was practical for this experiment. They are designed with

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<sup>32</sup> Hess, Manfred. "Paint Film Defects: Their Causes and Cure", Reinhold Publishing Corporation, New York, 1951. & Berger, Gustav A. and William H. Russell. "Conservation of Paintings: Research and Innovations", Archetype Publications: London, 2000.



guides that slide across the top of the substrate and lift the blade a uniform thickness above it. This blade pushes any excess paint down the length of a substrate, thus distributing a uniform layer of finish (see figures 9, 10 and 11). For the purposes of this experiment, more than 70 samples were needed and their small dimensions would not accommodate the common commercial blade design. After careful review of current products, a modified prototype was constructed, inspired by the Gardco Adjustable Micrometer "Microm" Film Applicator and the more simplistic Universal Blade Applicator.<sup>33</sup> The prototype illustrated in figure 8 proved successful for the purposes of this experiment.

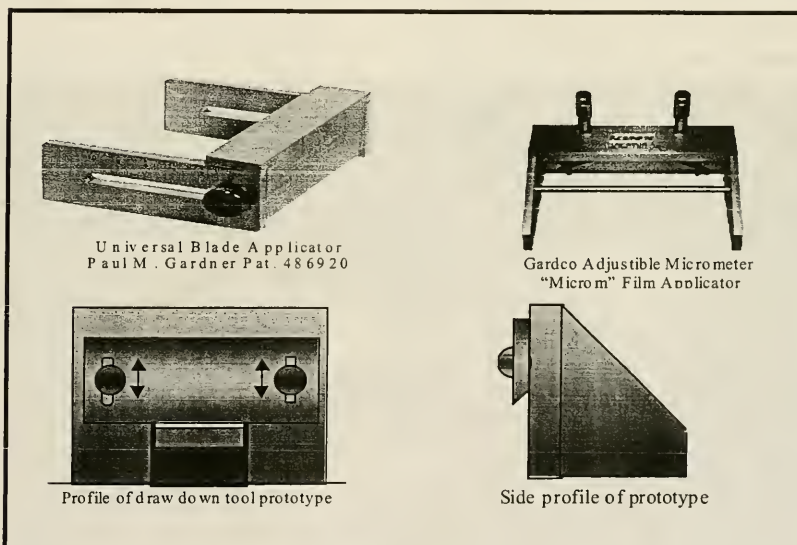


Figure 8. Gardner Company Inc. and author's prototype Draw Down Bar Applicators

<sup>33</sup> Paul M. Gardner Company Inc., "Testing Instruments 65 Years Anniversary Catalog", 1996-2002.  
<http://www.gardco.com/>



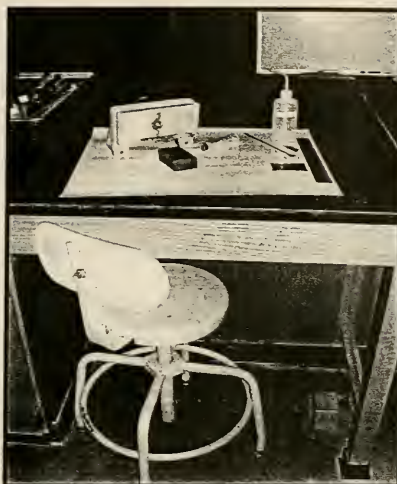


Figure 9. Tools used to apply reproduction earthen surface finishes



Figure 10. Reproduction earthen surface finishes before weathering and treatments



Figure 11. Author applying reproduction earthen surface finishes



## 6.1 Substrate Porosity

Four types of brick substrate were tested to determine their porosity. The same tests were run on fragments of unmodified sandstone obtained from the midden of site 5MV650 in Mesa Verde National Park. Assessment of substrate porosity was conducted in accordance with Experiment #8: Water Absorption by Total Immersion and Experiment 13: Porosity in Solids: Hygrostatic Weighing as specified in the ICCROM published ARC: A Laboratory Manual for Architectural Conservators.<sup>34</sup> Three samples of sandstone obtained in the vicinity of 5MV650 were tested for porosity. Three samples each of four additional substrates were also tested for their comparable porosity. Please see attachment A for detailed data on substrate porosity.

Based on the level of stone decay observed at site 5MV650, it was assumed that the porosity of exposed building stone would be high. However, the stone that was tested was not exceptionally porous. This may be partially attributed to the method of sample preparation for this test. In order to obtain consistent data for comparison to manmade materials, all samples were prepared in the same manner. Each sample was cut to exact specifications on a diamond bit rotary saw. This method of preparation removed the majority of weathered surface from the exterior of each facsimile masonry unit producing a smooth and uniform surface. The low porosity of recently modified sandstone and dry climate contributes to the durability of this ancestral architecture.

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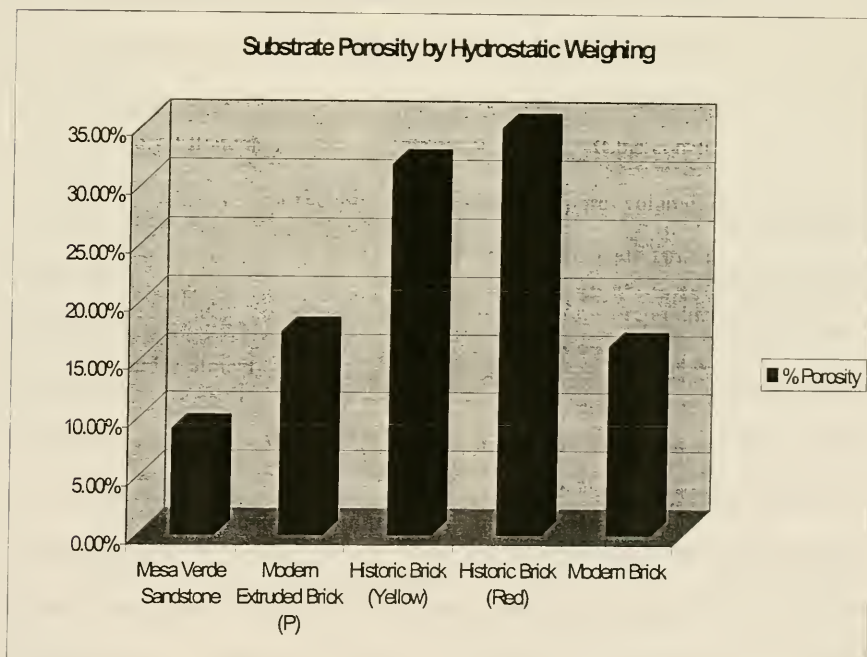
<sup>34</sup> Teutonico, Jean Marie. "ARC: A Laboratory Manual for Architectural Conservators", ICCROM, 1988, p. 35-40 and 52-55.





The porosity of sandstone fluctuates due to environmental exposure, pore shape and pore configuration. This variability can substantially affect the rate of deterioration of both the stone substrate and surface finishes applied to it. At the conclusion of this test, the facsimile substrate most closely resembling the pore volume of Mesa Verde Sandstone was selected as a substrate for further testing (see table 19). Thus, the modern brick was selected as a substrate for subsequent surface finish reproduction, accelerated weathering, and treatment.

**TABLE 19**  
**Porosity of substrate samples determined by Hydrostatic Weighing**





## 6.2 Relative Shrinkage of Reproduction Surface Finishes

A simple test was devised to observe the relative contraction of different reproduction plaster mixtures. This test was based on modern tests to determine the linear shrinkage for soils. Modern extruded bricks were cut to form a coupon with a surface measuring 100 mm. by 100 mm. Three mixtures were applied at a thickness of approximately 5 mm. in accordance with ASTM Test Method D4062-99 Standard Test Method for Leveling of Paints by Draw-Down Method.

### Reproduction Plaster Sample Mixtures:

- Reddish Brown Plaster = 60:40 sand to silt/clay ratio of SY-3 soil
- Reddish Brown Wash = 10:90 sand to silt/clay ratio of SY-3 soil
- White Sandstone Wash = 10:90 sand to silt ratio/clay of MR-1 soil

Each coated brick was placed in a desiccator for 120 hours with an atmospheric temperature of 20 degrees Celsius and 30% relative humidity. As further proof of the correlation between particle size, shrinkage and adhesion, the sample of reddish brown plaster remained fully adhered to its substrate while the white wash and reddish brown wash mixtures each pulled up from their substrate at the edges. Since this detachment made it difficult to measure shrinkage, both washes were re-set to their substrate by wetting with water by capillary action. Then, both were covered with a sheet of fibrous Japanese tissue paper tucked under the edges of each brick to prevent the wash from pulling up from its substrate as it dries. The washes were dried once again before



measurements were made to determine relative shrinkage (see figure 12 and table 20).

For more data on this experiment refer to Appendix F.

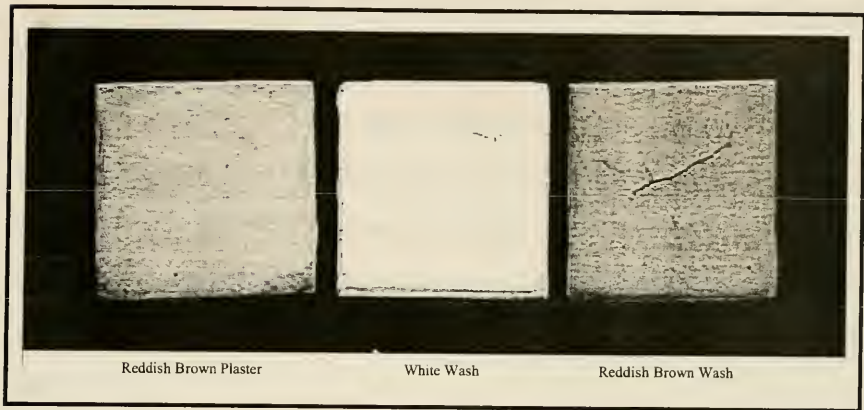


Figure 12. Dried Reproduction Surface Finish Samples

TABLE 20

Results of Test to Determine the Relative Shrinkage of Reproduction Finishes

Sample	Area (Dry)	% Shrinkage	Visual Observations
Reddish Brown Plaster	50000 mm.	Nominal	No cracks and no visible shrinkage
White Sandstone Wash	20958 mm.	58%	3 large cracks at concentrated at one corner
Reddish Brown Wash	39680 mm.	21%	1 large central crack and 14 smaller cracks

### 6.3 Relative Water Absorption of Reproduction Surface Finishes

Based on particle size analysis and previous documentation, three plaster mixtures were selected for subsequent testing. All plasters consisted of a 60:40 sand to silt ratio and all washes were made up of a 10:90 sand to silt ratio based on particle size. The



To reproduce the aesthetic effects observed in the field, different Mesa Verde soils were used to produce different colored washes.

Five reproduced plaster samples were used in this experiment to test the water absorption of similar finishes in situ (see figure 13). This procedure is described in Experiment #9: Water Drop Absorption as specified Teutonico (1988).<sup>35</sup> Additional measurements were made immediately after each drop of water was absorbed into the sample. The diameter of the water stain made by each drop was immediately measured as a relative indication of surface tension and spreadability of the solution. Temperature at the time of experiment was 70 degrees Fahrenheit and the Relative Humidity was 34-35%.

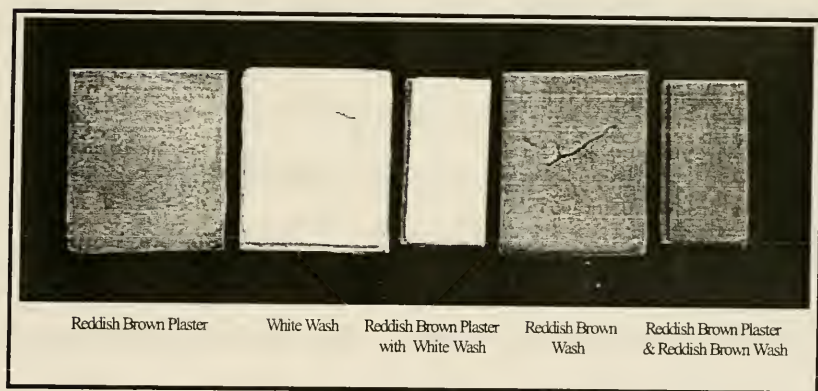


Figure 13. Surface Finish Samples used to Test Relative Absorption

<sup>35</sup> Teutonico, Jean Marie. "ARC: A Laboratory Manual for Architectural Conservators", ICCROM, 1988, p. 41-42.





### Reproduction Plaster Sample Mixtures:

- Reddish Brown Plaster = 60:40 sand to silt ratio of SY-3 soil
- Reddish Brown Wash = 10:90 sand to silt ratio of SY-3 soil
- White Sandstone Wash = 10:90 sand to silt ratio of MR-1 soil
- White Wash = 10:90 sand to silt ratio of MR-1 sand and SY-1 silt

The above tables record the absorption time in tenths of seconds for unweathered samples (UW) and for samples that have been subjected to accelerated weathering (W). Further measurements were taken to determine the difference between weathered and unweathered samples treated with different adhesives. Despite the rapid absorption rate of these samples, it can be concluded that weathered surface finishes are more absorptive than unweathered surface finishes. The white caliche washes that were treated and exposed to the same environmental conditions showed less fluctuation in absorption rate regardless of whether they were treated or not. The data generated by this experiment suggests that particle size distribution of the earthen finish layers may affect the ability of an adhesive to thoroughly wet the particles. Wettability of an adhesive affects both bond strength performance and reflectance. Consequently, an understanding of relative water absorption is influential when selecting an adhesive formulation to treat environmentally weathered earthen surface finishes.



## Chapter 7

### ACCELERATED WEATHERING – PRELIMINARY TESTING

This experiment was inspired by ASTM D4141-01 Standard Practice for Conducting Black Box and Solar Concentrating Exposures of Coatings. The experiment was structured to simulate the environmental factors affecting surface finish deterioration, yet these factors were simulated at a higher rate of fluctuation than is present in situ. Samples were subjected to controlled fluctuations of heat and moisture and monitored for evidence of adhesion loss between the substrate and finish layers.

#### 7.1 First Cycle

The first cycle attempted to reproduce blistering by drying a wet sample in the oven. This sample was applied to an extruded brick substrate with a porosity of 8.53% measured gravimetrically and 17.4% measured hygrostatically. The result was map cracking or hairline fractures.

#### 7.2 Second Cycle

The second cycle attempted to reproduce blistering by putting a wet sample in the oven within a dish of water to reproduce constant heat from the sides and top but rising damp from the bottom. This sample was applied to the Modern Brick substrate. The result was cracking through to substrate.

#### 7.3 Third Cycle

For the third cycle, four samples with different substrates were placed in a pan of water with a heat lamp on them and with repeated misting (approximately 6) for a two week period. Sample X was also cycled with this group of samples. Two weeks into the



experiment, each sample was tilted at a 45° angle while wet to see if such samples would be cohesive enough to subject subsequent samples to artificial weathering with a Q-U-V Accelerated Weathering Tester distributed by Q-Panel Company. This action made it apparent that surface finishes applied to the Historic Brick (Red) sample had fully detached from their substrate because both layers of surface finishes slid off the substrate in one clean action. At this point, the Mesa Verde Sandstone, Modern Extruded Brick (P) and Modern Brick samples were propped at a 45° angle and allowed to dry under heat lamps. Figure 14 shows the resulting cracks that extended through to the substrate.

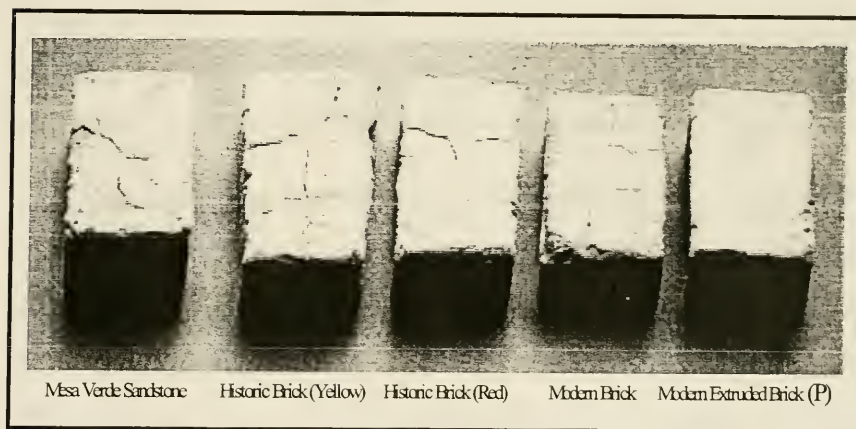


Figure 14. Samples used for preliminary testing of the accelerated weathering

Production of these cracks terminated the experiment for these samples until the Mesa Verde Sandstone was once again subjected to cyclic freezing, misting and heat lamps to see what the effect of moisture and wide temperature fluctuations would have on this sample. The result was interplanar cracking and blistering in close proximity to



already extant cracks (see table 21). Furthermore, the only examples of large scale blistering were observed for surface finish facsimiles prepared on Mesa Verde Sandstone substrates.

**TABLE 21**  
**Observations for Preliminary Accelerated Weathering Test**

<b>Sample</b>	<b>Observations</b>
Mesa Verde Sandstone	Moderate blistering and cracking to substrate when tilted at a 45° angle.
Modern Extruded Brick (P)	Minimal blistering and cracking to substrate when tilted at a 45° angle.
Modern Brick	Moderate blistering and cracking to substrate when tilted at a 45° angle.
Historic Brick (Red)	Moderate blistering and loss of substrate when tilted at a 45° angle.
Sample X	Large, single blister formed in the center of this sample between the substrate and the surface finishes.

Loss of adhesion between the substrate and surface finishes occurred differently for different types of substrates. For example, the historic brick sample exhibited a uniform loss of adhesion at the interface between substrate and finish. This is probably due to the high porosity of this substrate. The water quickly penetrated through the substrate but was unable to penetrate the less porous surface finishes at the same transmission rate. This differential rate of water transmission caused the water to congregate in a thin film between the substrate and surface finishes. This film was thick enough to actually detach and suspend the finish layers above the substrate, enabling the finish layers to slide off the substrate in a single motion when the sample was tipped at a 45 degree angle. Due to the low porosity of both modern brick samples, each displayed swelling and cracking of their surface finishes but neither sample exhibited an noticeable





loss of adhesion between substrate and surface finish. The two samples of Mesa Verde sandstone exhibited shrinking, swelling and blistering of their surface finishes. Sample X exhibited immediate and extensive blistering while the other sandstone substrate exhibited the smaller, salt covered blisters shown previously in figure 5. The presence of salts and the abundance of surface finish layers applied to sample X may account for the immediate and extensive blistering observed on this sample. Appendix J provides a photographic comparison of all samples tested in this experiment. The large central blister on Sample X has been broken in this photograph to show the extent of adhesion loss at the conclusion of this experiment.



## Chapter 8

### ACCELERATED WEATHERING OF REPRODUCTION SURFACE FINISHES

Fifty-six samples of reproduction surface finishes were created for this test. A brick with a gravimetric porosity of 7.5% and a hydrostatic porosity of 16.2 was applied with a 60:40 fine sand to silt and clay mixture of plaster and allowed to dry before applying a 10:90 mixture of wash on top of it. After all samples had cured for at least one week, they were subjected to accelerated weathering. Fifty-four samples were artificially weathered for a period of five weeks. At the end of each seven-day weathering cycle, the samples were fully dried and their conditions were mapped. Samples were cycled for a total of 12 hours a day for five cycles of seven days each. At the end of each seven-day cycle, the samples were allowed 48 hours of open-air exposure to fully dry before the condition of each sample was recorded according to ASTM D714-87 Standard Practice for Evaluating the Degree of Blistering of Paints, E1808-96 Standard Guide for Designing and Conducting Visual Experiments, D660-93(2000) Standard Test Method for Evaluating Degree of Checking of Exterior Paints and D661-93(2000) Standard Test Method for Evaluating Degree of Cracking of Exterior Paints. Each successive seven-day cycle was characterized by an incremental increase in the intensity of temperature and relative humidity fluctuations to which the samples were exposed. For each of the weathering cycles noted below, attempts were made to maintain room temperature at 24° Celsius with an RH of 25%. However, fluctuation in the ambient room temperature varied as much as 18° due to problems with the school heating system. The freezer was



set at -10° Celsius with an RH of 100%, and the oven was set at 40° Celsius with an RH of 20%.

### **8.1 First Cycle**

Samples were cycled for a seven-day period. For the entire 11-hour period, samples were placed under heat lamps and monitored for changes in temperature and relative humidity. At the start of this cycle, samples were placed in a shallow water bath and allowed to absorb water through capillary action for 1½ hours. Three hours into the cycle, the tops of the samples were misted with water. Six hours into the cycle, the samples were misted and placed in the 1½-hour shallow bath once again. Nine hours into the cycle, the tops of all samples were misted again. 11 hours into the cycle, the heat lamps were turned off and samples were allowed to air dry until the beginning of the next cycle, 12 hours later.

#### **Preliminary Observations:**

Minute interfacial blister formation on the caliche samples while wet. This effect is less visible once the samples have dried. This effect may not be the result of true blistering that is typically caused by isolated interfacial adhesive loss, but may be attributed to salt crystallization. Many samples have detached from their substrate.

### **8.2 Second Cycle**

Samples were cycled for a seven-day period. At the start of this cycle, frozen samples were placed under heat lamps and monitored for changes in temperature and relative humidity. Four hours later, tops of all samples were misted with water and then placed in the freezer for four hours. Upon removal from the freezer, samples were placed in a shallow water bath and placed under the heat lamps. Twelve hours after the start of



this cycle, the samples were once again placed into the freezer and allowed to remain frozen for 12 hours until the start of the next cycle.

#### Preliminary Observations:

Minor cracking of brownish red samples. No substantial change in the condition of caliche samples since the last monitoring. All samples have detached from their substrate.

### 8.3 Third Cycle

Samples were cycled for a seven-day period. At the start of this cycle, frozen samples were placed under heat lamps and covered with plastic containers to retain humidity as they warmed. Three hours later, the samples were placed in the freezer. Samples were removed from the freezer three hours later and placed under the heat lamps. Eight hours into this cycle, the samples were covered again to raise the relative humidity. Twelve hours into the cycle, samples were again placed into the freezer and remained frozen for 12 hours until the start of the next cycle.

#### Preliminary Observations:

Condensation induced minor water damage on a few samples when water drops fell onto them. Current conditions accelerating at a slow rate. As an aside, the sandstone sample used in the initial testing phase was further subjected to cycle 3 weathering. This sample was not detached from its substrate. Nor was it suffering large-scale interfacial detachment. Before the outset of cycle 3 weathering, this sample exhibited major cracking that was induced by rapidly drying out the sample. Cycle 3 accelerated weathering of this sandstone sample resulted in significant blistering concentrated around the major cracks in the finishes. By observing the rapid change in condition exhibited by





this sample as compared to the lack of change in the rest of the samples, it was determined that inducing rapid drying would accelerate the deterioration of all samples.

#### **8.4 Fourth Cycle**

Samples were cycled for a seven-day period. At the start of this cycle, frozen samples were placed in an oven for three hours. The top of each sample was misted with water and placed in to the freezer. Three hours later, the samples are returned to the oven. After three hours in the oven, samples are misted once again and placed in the freezer until the beginning of the next cycle.

##### **Preliminary Observations:**

Rapid drying and major temperature fluctuations induced by oven drying resulted in major cracking of the reddish brown samples. Yet the caliche samples remain unchanged excepting a few samples where the edges of the caliche layer curled up due to contact with the oven wall. Now that cracking had been induced, the samples were once again weathered according to the specifications of the third cycle.

#### **8.5 Fifth Cycle**

Samples were cycled for a seven-day period in accordance with the procedure set forth for cycle 3.

##### **Preliminary Observations:**

Major cracking and interfacial delamination has occurred for the reddish brown samples. Yet, the caliche samples did not exhibit major cracking. Desiccation of the earthen finish did however make them quite fragile and the precipitation of salts was evident on the surface of finishes when dry.



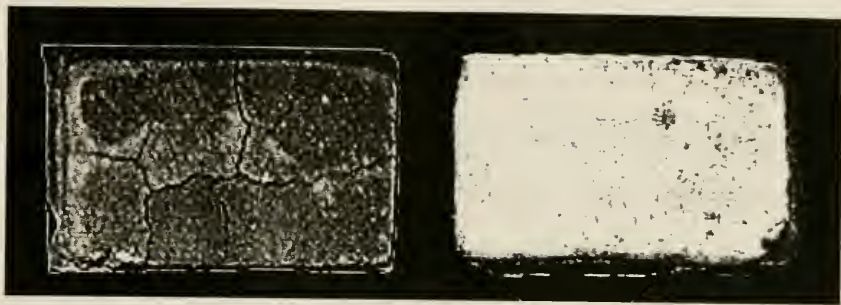


Figure 15. Accelerated weathering affects on facsimile reddish brown soil and white calcium carbonate finish

Cracked finishes occur as a result of moisture induced swelling and thermal contraction of the finish layer while being restrained by the rigid support beneath it.<sup>36</sup> At the conclusion of this experiment, samples were visually examined and their conditions recorded. There was a notable difference between the deterioration of reddish brown samples and that of white caliche samples. Reddish brown samples exhibited more expansion/contraction cracking and more interfacial delamination when exposed to the same temperature and relative humidity fluctuations than their caliche counterparts. This is attributed to the greater reactivity of clays when hydrated. It is also due to the high percentage of Calcium Carbonate in the white samples. Ground caliche particles consist of a nearly 70% calcium carbonate. Calcium carbonate forms crystals when hydrated. Thus, even caliche that has been ground fine enough to qualify within the silt and clay range of particle size is more rigid than the clay bound finishes.

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<sup>36</sup> Karpowicz, Adam. "A Study on the Development of Cracks on Paintings", Journal of the American Institute for Conservation, Volume 29, Number 2, Article 5, 1990, p. 169-180.



Clays, such the kaolinite found at Mesa Verde, are activated with water because the water coats each clay particle with a thin film of water that is electromagnetically held to the surface of the clay particle. This thin film of water acts as a lubricant between the particles of clay, allowing the finish layer to expand. In this hydrated state, the clays are more flexible than when they are dry. The amount and rate of expansion depends on the amount of clay particles within the finish formulation. When the finish layer is dried rapidly, the particles do not have time to return to their original position before this thin film of water evaporates.

With the reddish brown samples, differential expansion and contraction of the plaster and wash layers produced consistent failure. The greater shrinkage of the wash layer in comparison to its underlying plaster layer and the stronger adhesion between the two finish layers as compared to adhesion between the finishes and their substrate caused the finishes to lift up off the substrate. A build up of stress induced by this differential shrinkage concentrates strain at the center of the surface finish. Cracking at the center of the sample releases this build up of internal stress within finish layers (see figure16). However, it also causes a weakness in the cohesion of the layer and allows water to penetrate the crack. Freezing of water within such cracks causes further stress due to the expansion of the frozen water placing pressure onto the surface finishes. Thus, a cracked surface finish deteriorates faster than one that exhibits no cracking.



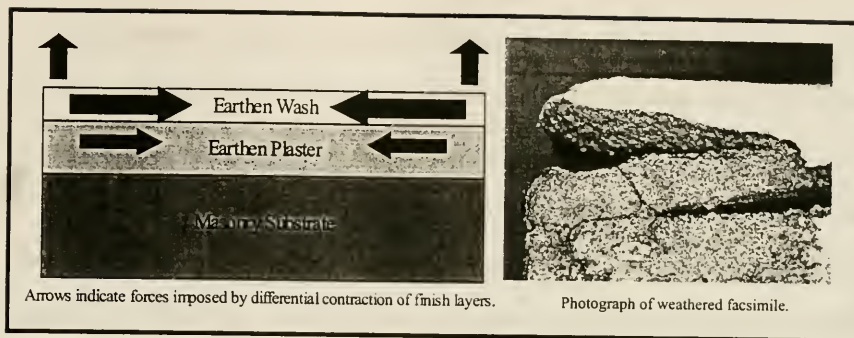


Figure 16. Delamination and detachment of facsimile earthen surface finishes

In contrast to the wash formulated with reddish brown soil, two different types of white binder were formulated. Both binders were made by grinding stone to a particle size that was small enough to pass through a 75 $\mu$ m screen. Each was then mixed with the appropriate portion of sand sized particles and applied to the same substrate as the reddish brown samples. Even before subjecting these samples to accelerated weathering, it was apparent that the white sandstone binder shrank much more than the caliche binder. In fact, five of the twelve samples made from white sandstone cracked while drying after the wash was applied. The process of grinding white sandstone small enough to pass through a 75 $\mu$ m screen is not an exacting method and probably ground the particles smaller than was needed to clear the screen. Theoretically, the smaller the particles, the more that a finish layer will shrink as it dries. However, the caliche samples were made according to this same method and since none of the 28 caliche samples exhibited this extent of cracking even after weathering.





The cohesive strength of both wash formulations are dependant upon the amount of surface area that particles within the finish are in contact with each other. When wet, the surface area of each particle is in complete contact with the water making the water act as a binder in the clay formulation while it is wet. When dry, both the caliche and clay particles are small enough that they fill the voids between the sand particles. The application of finishes by smoothing them onto their substrate both compresses and aligns the particles parallel to the surface of their substrate. This interlocking of particles accounts for the cohesive strength within a finish layer and make finishes with a high clay content less porous than those formulated with a high sand content. Washes with high clay content characteristically swell more when hydrated and are more cohesive when applied in thin layers.



## Chapter 9

### BINDING AGENTS COMMONLY USED IN ADHESIVE, SEALANT, AND PAINT FORMULATIONS

The following text lists some of the traditional binders used in modern paintings. This brief overview of paint binders does not go into detail regarding specific types of materials used to formulate alkyd or proteinacious binders. Since the subject of this paper is to compare the material properties of two formulations of acrylic and two formulations of protein based adhesives, the following description only provides an overview of potential alternatives.

#### 9.1 Mineral Binders

For the purposes of this paper, binders formed by crystalline formation and particle size distributions that produce a strong enough intermolecular attraction to produce a stable film will be classified as mineral binders. Finishes in which the pigment or filler particles are bound by crystal formation include mortars, cements, frescos, calcium based plasters and some stuccos. These are reaction adhesives that form strong secondary bonds that exhibit minimal shrinkage and a rigid structure.<sup>37</sup> Using the example of fresco painting, pigment is bound in place by a chemical reaction that occurs when the applied suspension of wet calcium hydroxide and fine sand reacts with the air to form a crystalline calcium carbonate matrix that secures the pigment particles into place. During this process, quicklime that is formed by heating calcium carbonate, and then hydrated to

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<sup>37</sup> Wilks, Helen et al. Science for Conservators: Adhesives and Coatings, Volume 3, Conservation Unit of the Museums & Galleries Commission and Routledge, 1992.



form lime, binds the fresco pigments with the crystal structures that form as the lime cures and reacts with the air. Aluminum and potassium silicates are added to plaster to form cements<sup>38</sup>

Due to their structure, calcium carbonate particles expand and contract with water less than comparably sized clay particles. This property and the white color of calcium carbonate make derivatives such as chalk, whiting, lime, caliche and gypsum are ideal for use as fillers and pigments for paint and plaster formulations. Mesa Verde soils that were tested potentially contain as much as 70% calcium carbonate. It is probable that these caliche, high lime content soils were used for making the plasters and mortars at Mesa Verde National Park.

## **9.2 Plant Binders**

Plant resins, gums and vegetable oils have been historically used in the formulation of paint binders as well. Organic varnishes, watercolors and oil paints are examples of plant binding mediums. The most common oil paint uses linseed oil obtained by processing flaxseeds. Film formation of linseed-oil-based paints occurs by the cross-linking of carbon-carbon double bonds that occurs as the film oxidizes.<sup>39</sup> Many plant binders are cellulose based.<sup>40</sup> Cellulose is a long chain polymer that occurs naturally in

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<sup>38</sup> Jusko, Don A. "History of Painting Mediums... Glue, Wax Paint, Cera Colla, Mastic, Casein Paint, Fresco, Egg, Oil Paint, Acrylic Paint", mauigateway.com.

<http://www.mauigateway.com/~donjusko/1mediums.htm#100,000 B/C>

<sup>39</sup> Dunn, Kevin M. "Paint", Course readings for CHEM710: Chemistry and Art, Hampden-Sydney College Department of Chemistry, 1996. <http://cator.hsc.edu/~mollusk/ChemArt/paint/paints.html>

<sup>40</sup> Wilks, Helen et al. Science for Conservators: Adhesives and Coatings, Volume 3, Conservation Unit of the Museums & Galleries Commission and Routledge, 1992.



plants.<sup>41</sup> Many plant-based binders were available to ancestral Puebloan painters.<sup>42</sup> For example, the pinion tree extruded a sticky sap that is still used today as a traditional sealant for ceramic vessels.<sup>43</sup> Boiling of the seeds also produces pine nut oil. Tree resins are the source of many alcohol and turpentine based binding systems whose use has been dated as early as 1600BC.<sup>44</sup> Archeologists have also found numerous vessels that have been repaired with an adhesive made from pinion pitch.(Figure 1.) Some modern Puebloan people still use pine pitch as a sealant for ceramic vessels used in traditional ceremonies.<sup>45</sup> Pine pitch is concentrated tree sap. Processing the sap of trees forms resins, distilled turpentine solvents, and fermented alcohols to dilute them.<sup>46</sup> Gum Arabic, a commercially processed tree sap, is combined with glycerin in the formulation of watercolor paint. It is also used in the formulation of gouache by combining gum arabic with powdered gypsum ( $\text{CaSO}_4$ ).

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<sup>41</sup> Wilks, Helen et al. *Science for Conservators: Adhesives and Coatings*, Volume 3, Conservation Unit of the Museums & Galleries Commission and Routledge, 1992.

<sup>42</sup> Beas, Maria Isabel G. "Traditional Architectural Renders on Earthen Surfaces", University of Pennsylvania Masters Thesis, 1991

<sup>43</sup> Personal communication with archeologists and with staff from the CU Cultural Center in Cortez, CO, 10/15/2001.

<sup>44</sup> Jusko, Don A. "History of Painting Mediums... Glue, Wax Paint, Cera Colla, Mastic, Casein Paint, Fresco, Egg, Oil Paint, Acrylic Paint", [mauigateway.com](http://www.mauigateway.com/~donjusko/1mediums.htm#100,000%20B/C).

[http://www.mauigateway.com/~donjusko/1mediums.htm#100,000 B/C](http://www.mauigateway.com/~donjusko/1mediums.htm#100,000%20B/C)

<sup>45</sup> Artifact Database for the Chappell Collection of ceramic vessels stored at the Anasazi Heritage Center.

<sup>46</sup> Jusko, Don A. "History of Painting Mediums... Glue, Wax Paint, Cera Colla, Mastic, Casein Paint, Fresco, Egg, Oil Paint, Acrylic Paint", [mauigateway.com](http://www.mauigateway.com/~donjusko/1mediums.htm#100,000%20B/C).

[http://www.mauigateway.com/~donjusko/1mediums.htm#100,000 B/C](http://www.mauigateway.com/~donjusko/1mediums.htm#100,000%20B/C)





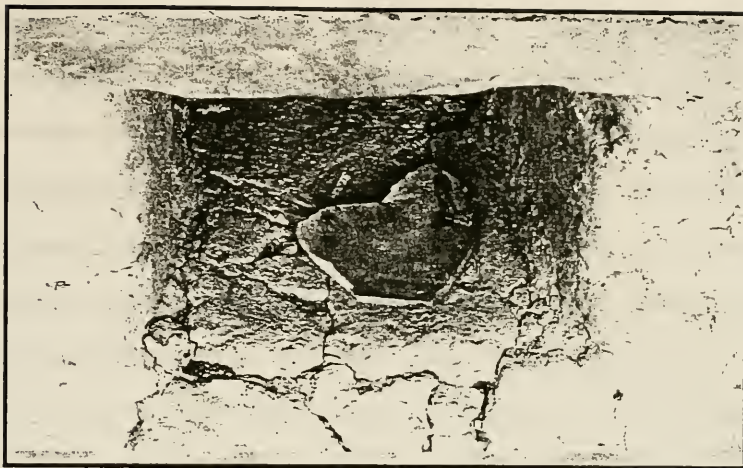


Figure 17. Ancestral Puebloan sherd mended with pinion pitch.  
Found by Archeologist Joel Brisben in Room 16(1) of site 5MV640, July 2002

### 9.3 Wax Binders

Common waxes used as paint binders and adhesives include beeswax, paraffin wax and more recently, microcrystine wax. Both beeswax and petroleum based waxes are thermoset polymers that primarily consist of hydrocarbons. Beeswax was commonly used as a binder in ancient Egypt, Greece and Rome into the 8<sup>th</sup> century AD.<sup>47</sup> Waxes are still used by modern day painters and some traditional fresco painters to enhance their artworks with overpainted detailing.<sup>48</sup>

### 9.4 Alkyd Binders

Alkyds are manmade formulations of polymeric esters that cross-link during film formation. This classification doesn't fit neatly into the classification of animal, plant or

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<sup>47</sup> <http://cator.hsc.edu/~mollusk/ChemArt/paint/paints.html>

<sup>48</sup> Personal communication with Richard Raisellis



mineral since the oils used in alkyd formulations may be derived from any of these three initial sources. The value of saturation for an oil determines its chemical properties as it approaches its glass transition point. Commercially, alkyd paints are classified by the amount of oil contained within the solution. These classifications vary from Type I oil-free polyester to Type IV long oil alkyds that contain more oil than is able to react within the molecule through esterification.<sup>49</sup> Many oils used in the production of alkyd paints are also used as plasticizer additives within the formulation of paints based on other mediums. It is notable that alkyd paints are often recommended for alkaline surfaces due to the tendency of the more acidic formulations to saponify.<sup>50</sup> However, the inclusion of phenolic resin additives can improve an alkyd paints alkali resistance.<sup>51</sup>

The most common polyol used in the manufacture of alkyd paints is glycerin. Glycerin is obtained as a by-product of soap manufacture and may be of either animal or vegetable origin. In the early 1940s glycerin was able to be commercially produced from petroleum sources.<sup>52</sup> Glycerin has a high boiling point and a lower viscosity than other polyols with a functionality higher than three. This polyol is worthy of note for this paper since glycerin is currently being used as a plasticizer in the gelatin-based treatment adhesive used at Mesa Verde.

One other polyol sometimes used in the production of Alkyd paints should be noted. Sorbitol is commonly used as a plasticizer for gelatin in the food industry.<sup>53</sup> It is

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<sup>49</sup> Martins. Alkyd Resins p29

<sup>50</sup> Hess, Paint film defects

<sup>51</sup> Martins. Alkyd Resins p85

<sup>52</sup> Martins. Alkyd Resins p40

<sup>53</sup> SORBITOL



“produced by the catalytic hydrogenation of glucose.”<sup>54</sup> Sorbitol contains six hydroxyl groups but is usually referenced as a functionality of four due to the inability of all hydroxyl groups to esterify by normal alkyd processing techniques.

### **9.5 Acrylic Binders**

Since 1945, manmade acrylic esters have been manufactured as a paint medium. Both pigment and monomers of compounds such as methyl acrylate or vinyl acetate are suspended within an aqueous solution.<sup>55</sup> Depending on the chemical formulation of the acrylic solution, film formation may occur through either solvent loss or by crosslinking of these monomers. Acrylics are commonly used as adhesives and sealants for artifact and painting consolidation. Reasons for their use include consistencies in commercial manufacture, well-established solubility parameters and transparency of the resulting film. The most common, modern day, archeological use of acrylic resins is the use of Acryloid B-72 (US. Patent) / Paraloid B-72 (UK. Patent) for artifact labeling and mending potsherds. The National Park Service has used Rhoplex brand acrylic emulsion modified soil mortars to stabilize structures at archeological sites since the early 1970s.<sup>56</sup> In fact, modified mortar and construction sealant formulations were sought by the National Park Service as early as the 1930s. Currently, Rohm and Haas Corporation supplies Mesa Verde National Park with Rhoplex E-330 for use in their acrylic emulsion

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<sup>54</sup> rtins. Alkyd Resins p43

<sup>55</sup> Dunn, Kevin M. “Paint”, Course readings for CHEM710: Chemistry and Art, Hampden-Sydney College Department of Chemistry, 1996. <http://cator.hsc.edu/~mollusk/ChemArt/paint/paints.html>

<sup>56</sup> Hartzler, Robert. “Acrylic-modified earthen mortar: a program of investigation and laboratory research into acrylic-modified earthen mortar used at three prehistoric Pueblo sites”, Conservation Program, Intermountain Cultural Resource Center, Intermountain Field Area, National Park Service, Dept. of the Interior, Santa Fe, NM, 1996, p. 18.



modified soil mortars.<sup>57</sup> Both of the acrylic emulsions selected for this study are marketed for the modification of soil mortars by Rohm and Haas Corporation. Numerous adhesives such as methyl methacrylates, silicone solutions and polyvinyl acetates have also been used as adhesives to stabilize earthen plasters at archeological sites.

## **9.6 Animal Binders**

Often credited as the first paint binder used, animal proteins such as milk, egg, blood and gelatin are still used today. Proteins are co-polymers made up of amino acids.<sup>58</sup> The subgroup Albumin includes “any of numerous simple heat-coagulable water-soluble proteins that occur in blood plasma or serum, muscle, eggs, milk, and other animal substances and in many plant tissues and fluids.”<sup>59</sup> When used as film forming binders, this group of proteins is procured through the denaturation of collagen.<sup>60</sup> These proteins are usually specified by the generic names given to the paint formulations that they are contained within.

Eggs are used in tempura formulations that were most common in European fine arts painting prior to the prominence of oil paints in the 15<sup>th</sup> century<sup>61</sup>. The properties of tempura film formation is attributed to both denaturation of protein and polymerization of fats within the egg yolk. “The proteins form many hydrogen bonds with each other and

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<sup>57</sup> Personal communication with Kathy Fiero, Stabilization Archeologist at Mesa Verde, 3/15/2002.

<sup>58</sup> Wilks, Helen et al. *Science for Conservators: Adhesives and Coatings*, Volume 3, Conservation Unit of the Museums & Galleries Commission and Routledge, 1992.

<sup>59</sup> Merriam-Webster Inc. “Dictionary”, <http://www.webster.com>

<sup>60</sup> Wilks, Helen et al. *Science for Conservators: Adhesives and Coatings*, Volume 3, Conservation Unit of the Museums & Galleries Commission and Routledge, 1992.

<sup>61</sup> Jusko, Don A. “History of Painting Mediums... Glue, Wax Paint, Cera Colla, Mastic, Casein Paint, Fresco, Egg, Oil Paint, Acrylic Paint”, [mauigateway.com](http://www.mauigateway.com/~donjusko/1mediums.htm#100,000 B/C).  
<http://www.mauigateway.com/~donjusko/1mediums.htm#100,000 B/C>





with the surface, locking the pigments into a solid matrix. As they age, these proteins form covalent bonds with each other, making the matrix very stable.”<sup>62</sup>

Milk is the common binder for casein. However, the term casein, or milk paint, has also been used to refer to paint formulations based on other protienacious binders. A survey of the literature revealed the use of gelatin and raw blood as binders used in the manufacture of casein.

Animal proteins such as gelatin and blood are used in both paint and adhesive formulations. For example, one early documented albumen solution formulated with a protein binder of egg whites and ox blood with a filler of lime was used as a construction sealant<sup>63</sup>.

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<sup>62</sup> Dunn, Kevin M. “Paint”, Course readings for CHEM710: Chemistry and Art, Hampden-Sydney College Department of Chemistry, 1996. <http://cator.hsc.edu/~mollusk/ChemArt/paint/paints.html>

<sup>63</sup> Thornton, Jonathan. “A Brief History and Review of the Early Practice and Materials of Gap-Filling in the West”, *Journal of American Institute for Conservation* 1998, Volume 37, Number 1, Article 2, p. 3-22.



### GELATIN BASED ADHESIVES

Gelatin is an animal binder derived from animal proteins. It is generally defined as a more refined version of what artists term hide glue. Hide glue has been traditionally combined with calcium carbonate or calcium sulfate and water in formulations for sizing, gesso, gauche used in fine arts applications. Furthermore, calcium carbonate in the form of chalk or whiting has served as a traditional paint pigment in paint formulations utilizing various other mediums as well. Gelatin and calcium carbonate have been combined with plant resins to formulate architectural composition reliefs. Gelatin also has a longstanding history of use as photographic emulsions. Weak protein solutions are also used as biodegradable-facing adhesive when administering conservation treatments on calcium carbonate surfaces.<sup>64</sup>

There are many different forms of animal based glue with the forms distinguished by which animal parts are used for its manufacture and the way that the glue is processed.(Figure 2.) The type of animal, parts of the animal used, and age of the animal can all affect physical properties of the final product even when a product is marketed under the same name. Common fluctuations in material properties include differences in pH and gel strength. Animal glue collagen is obtained from the bone, tendon and skin of

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<sup>64</sup> Berger, Gustav A. and William H. Russell. "Conservation of Paintings : Research and Innovations", Archetype Publications: London, 2000.



animals. Other naturally occurring polymers are obtained by processing the keratin that is contained within feathers, horns and hair.<sup>65</sup>

**TABLE 22**  
**Types of Commercially Available Gelatin**

Type of Gelatin	Raw Material	Physical Properties
Isnglass	sturgeon bladders	Low gel point
Hide glue	bovine hide	High coefficient of expansion
Bone glue	bovine bones	Moderate coefficient of expansion
Animal glue	bovine bones, bovine hide, and pig skin	High coefficient of expansion
Rabbit skin glue	Rabbit skin	High molecular weight
Fish glue	Fish skin	Weak gel strength

Gelatin is an organic, hydrocolloidal compound of proteins that is produced from the denaturation of animal collagen. The chemical composition of gelatin-based hide glue roughly constitutes 51% carbon, 6% hydrogen, 24% oxygen and 18% nitrogen.<sup>66</sup> Historically, animal glues have been made by breaking the original polymer chains derived from animal hides, tendons and bones. This was done by boiling the animal parts at a temperature of 80-90° Celsius<sup>67</sup> to break their naturally occurring polymer chains and

<sup>65</sup> Wilks, Helen et al. *Science for Conservators: Adhesives and Coatings, Volume 3*, Conservation Unit of the Museums & Galleries Commission and Routledge, 1992.

<sup>66</sup> Matt T. Roberts, Don Etherington and Margaret R. Brown. "Bookbinding and the Conservation of books: A Dictionary of Descriptive Terminology", Stanford University Libraries, 1994.  
<http://palimpsest.stanford.edu/don/don.html>

<sup>67</sup> Matt T. Roberts, Don Etherington and Margaret R. Brown. "Bookbinding and the Conservation of books: A Dictionary of Descriptive Terminology", Stanford University Libraries, 1994.



then concentrating the resulting broth<sup>68</sup> by drying it into sheets of solid gelatin. However, the denaturation of proteins can be induced through many different methods. Exposure to heat, pressure, salts, acids, alkalis, and alcohols can be used for the denaturation of collagen. The production of gelatin involves two chemical modifications. The first is to induce the denaturation of the proteins while the second is a process of aggregation.<sup>69</sup>

Modern methods for mass-producing gelatin use acid or alkaline digestion of animal parts to rapidly break these chains. The resulting gelatin differs in pH and viscosity depending on the raw materials used and manufacturing process. The grade of the final product is largely dependant upon what type of animal parts are used, the age of the animals<sup>70</sup>, the purity of processing through filtering. Demineralization to remove salts and the extent to which fats were originally removed also affects the physical properties of gelatin. The pH of gelatin is largely determined by its manufacturing process. Hide glues generally have a pH within the range of 6.5-7.4. Acidic glues tend to absorb less water and set more slowly than glues that are more alkaline. Gelatin manufactured by means of an acid bath generally has an isoionic point between 7 and 9 while alkaline processed gelatin ranges between 4.8 and 5.2.<sup>71</sup> Gelatin has “both positive and negative charges on the molecule (and no net charge at the isoionic point).”<sup>72</sup> This allows the gelatin to crosslink with acid groups when the gelatin is at a pH that enables

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<http://palimpsest.stanford.edu/don/don.html> & Cole, Bernard. “Gelatine Food Science”, University of Pretoria, <http://www.gelatin.co.za/gltln1.html>

<sup>68</sup> Thornton, Jonathan. “A Brief History and Review of the Early Practice and Materials of Gap-Filling in the West”, *Journal of American Institute for Conservation* 1998, Volume 37, Number 1, Article 2, p. 3-22.

<sup>69</sup> Gossett, P.W., S.S.H. Rizvi, and R.C. Baker. “Quantitative Analysis of Gelation in Egg Protein Systems”, *Food Technology*, Vol. 38, Num. 5, May 1984, p. 67-96.

<sup>70</sup> Cole, Bernard. “Gelatine Food Science”, University of Pretoria, <http://www.gelatin.co.za/gltln1.html>

<sup>71</sup> Cole, Bernard. “Gelatine Food Science”, University of Pretoria, <http://www.gelatin.co.za/gltln1.html>

<sup>72</sup> Cole, Bernard. “Gelatine Food Science”, University of Pretoria, <http://www.gelatin.co.za/gltln1.html>





the side chains to not carry an electrostatic charge.<sup>73</sup> Thus, pH affects the stability, solubility<sup>74</sup> and potential for internal plasticity when combined in an adhesive solution. "A gel network with a certain degree of order can be attained if the aggregation step occurs more slowly than the denaturation step. Thus giving the denatured protein molecule, time to orient themselves before aggregation; this is lower in opacity and higher in elasticity than one where aggregation is not suppressed. Schmidt (1981) suggested that if aggregation occurs simultaneously with denaturation, an opaque, less elastic gel results."<sup>75</sup>

As Matt T. Roberts and Don Etherington state, "Electrostatic Charge is one of the most commonly investigated factors. The pH as well as the ionic strength of the protein environment can alter the charge distribution among the amino acid side chains and can either decrease or increase the protein-protein interaction,. Nakamura et al. (1978) concluded that the main factor contributing to the heat-induced aggregation of ovalbumin (pI 4.5-4.6) is the degree of electrostatic repulsion among the denatured protein molecules. When the heat-denatured protein concentration is high (>0.5%), the aggregate size decreases as the pH increases from 5.8 to 10.0; this is due to increased repulsive forces among the protein molecules at the alkaline pH levels. Conversely, decreasing the pH or adding cations decreases the negative charge and accelerates aggregate formation, as does increasing the ionic strength."

Gelatins are thermoplastic long-chain polymers of collagen that have been broken into smaller chains attached with weak secondary bonds to molecules. The protein

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<sup>73</sup> Cole, Bernard. "Gelatine Food Science", University of Pretoria, <http://www.gelatin.co.za/gltnl.html>

<sup>74</sup> Cole, Bernard. "Gelatine Food Science", University of Pretoria, <http://www.gelatin.co.za/gltnl.html>



structure of gelatin is a triple helix structure held together by non-covalent molecular forces. The triple helix structure is formed from peptide bonded alpha chains of glycine, proline and hydroxyproline.<sup>76</sup> The content of amino acids within gelatin also includes glutamic acid, alanine, arginine, aspartic acid, lysine, serine, leucine, valine, phenylalanine, threonine, isoleucine, hydroxylysine, methionine, histidine and tyrosine depending on the method of manufacture.<sup>77</sup>

Gelatin is an organic polymer that is used in the production of adhesives (wood glues, medical adhesives), paints (distempers, canvas sizing, stucco marble), medical (surgical adhesive, thin-section microscopy samples, etc.), pharmaceutical drugs (gelatin capsules), foods, etc. Gelatin is manufactured to a more pure grade than traditional hide glues, but is essentially a variation of the same material.<sup>78</sup>

Numerous conservation studies have been conducted to assess the material properties of gelatin used in the manufacture of photographic emulsions and in the sizing and gesso of oil paintings. One question repeatedly addressed in these conservation studies include relative humidity induced expansion and contraction. Similar to the alignment of clay particles within a finish layer, fibrils within a gelatin solution naturally align themselves parallel to their substrate if the dwell time is long enough for them to do so. Above their glass transition temperature, gelatin solutions are viscous, amorphously structured solutions. This is because at high temperatures, their molecules slide over one

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<sup>75</sup> Gossett, P.W., S.S.H. Rizvi, and R.C. Baker. "Quantitative Analysis of Gelation in Egg Protein Systems", *Food Technology*, Volume 38, Number 5, May 1984, p. 67-96.

<sup>76</sup> Friedli, Georges-Louis. "Protien", Friedli Enterprises.

<http://www.friedli.com/herbs/phytochem/proteins.html#gelation>

<sup>77</sup> Cole, Bernard. "Gelatine Food Science", University of Pretoria, <http://www.gelatin.co.za/gltm1.html> (



another easily making the solution less viscous. The rigidity of a gelatin solution in gel state is determined by the spacing between cross-links along the molecule.

When the gelatin fibrils are aligned parallel to one another, hygroscopic swelling occurs primarily along one axis. Therefore, the way that a gelatin film swells in humid conditions is an indication of how well the fibrils are aligned to one another. It is significant that aligned fibrils are isotropic and misaligned fibrils exhibit birefringence when viewed microscopically. Studies conducted by Karpowicz, Simms and Blake have confirmed that the misalignment of these fibrils occurs when the film is restrained while curing.<sup>79</sup>

The method of adhesive application that is used at Mesa Verde would theoretically restrain the expansion and contraction of these fibrils unless there is sufficient time for the solution to cure. Another reason to allow for increased dwell time before the solution fully cures is to allow the adhesive solution to fully wet the particles of the substrate and finish.<sup>80</sup>

Previously published tests showed the same results as were exhibited with the cohesive shrinkage tests conducted for this study. On previous tests, the gelatin size remained intact at the center of the canvas, but cohesive shrinkage was such that the gelatin pulled into the center and damage resulted on the edges of the canvas due to this

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<sup>78</sup> Cassar, JoAnn and Roberta de Angelis. "Glossary. Materials used in 19th and 20th century Plaster Architecture. Plaster Architecture Essay, Culture 2000 Programme of the European Union. <http://www.plasterarc.net/essay/essay/CassarG.html>

<sup>79</sup> Karpowicz, Adam. "A Study on the Development of Cracks on Paintings", Journal of the American Institute for Conservation, Volume 29, Number 2, Article 5, 1990, p. 169-180.

<sup>80</sup> Berger, Gustav A. and William H. Russell. "Conservation of Paintings : Research and Innovations", Archetype Publications: London, 2000.



internal contraction.<sup>81</sup> Gelatin experiences a period of relaxation when exposed to a constant source of stress. In other words, gelatin that has been subjected to a constant level of stress may adjust to that level of stress rather than releasing the stress in the form of strain-induced cracks. This has been the topic of research in the storage of photographic emulsions and their reaction to fluctuations in relative humidity.

There are many uses of gelatin, so many additives are used when formulating it for those uses (see appendix D) For the purposes of this discussion, I will concentrate on additives that change the hardness, flexibility, biodeterioration resistance, viscosity and particle wettability. Each of these properties are relevant to the effectiveness and durability of in-situ reattachment. The contraction of gelatin solutions while approaching their glass transition temperature is so great that gelatin films are known to break ceramic and glass containers when heated for quick set in the lab.<sup>82</sup> The pharmaceutical, food and photographic industries have identified many cross-linking polymers that join with gelatin and often serve more than one function. Common additives that change the elasticity (E-modulus) of gelatin solutions include polyhydric alcohols like glycerol, propylene glycol, sorbitol, and formaldehyde. Cross-linking with acrylic is being studied and cross-linking of gelatin with aldehydes such as glutaraldehyde is also being researched. Cross-linking of gelatin using the enzyme trans-glutaminase is used to join gelatin to other proteins.

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<sup>81</sup> Karpowicz, Adam. "A Study on the Development of Cracks on Paintings", *Journal of the American Institute for Conservation*, Volume 29, Number 2, Article 5, 1990, p. 169-180.

<sup>82</sup> Cole, Bernard. "Gelatine Food Science", University of Pretoria, <http://www.gelatin.co.za/gltm1.html>





### CURRENT ADHESIVE TREATMENT METHODS

The following treatment description outlines the treatment methods developed by the University of Pennsylvania Architectural Conservation Lab. This method addressed in this study uses different concentrations of Gelatin in solution applied at different temperatures. Since gelatin can be converted to three different states based on its water content and temperature, three methods of application were developed for the use of this adhesive.

#### Adhesive Formulations:

(percentages of weight to volume)

- 1) 5% gelatin plus 10% glycerin in water
- 2) 10% gelatin plus 10% glycerin in water

Prior to all treatments, the area of detachment is fully documented, cleared of all loose material and debris that may have collected behind the layer of surface finish, and pre-wetted with water or water and isopropyl alcohol. This initial preparation is very important to the longevity and adhesive strength of any treatment. Once the adhesive is applied, the exterior surface of the detached layer is misted with a solution of 30% isopropyl alcohol in water. The alcohol is a wetting agent, which lowers the surface tension of the water allowing the water to more readily coat the particles that make up the detached layer. Both the glycerin that is used in the formulation of these gelatin solutions



and the isopropyl alcohol serve as wetting agents in the production of printing fountain solutions.<sup>83</sup>

Glycerin is an oil that can be obtained from many substances. Glycerol of the chemical formula  $\text{CH}_2(\text{OH})\text{CH}(\text{OH})\text{CH}_2\text{OH}$  is a trihedic alcohol with a molecular weight of 92.09 and a specific gravity of 1.26. It is processed from petroleum products, vegetables and animals. Its most common use is in cosmetics and soaps. As a surfactant, it lowers the surface tension of water at the interface of water to solid. In the case of pigment particles, this allows the particles to be more thoroughly coated with the binder. It is termed a surfactant and a wetting agent when used in this fashion.

Glycerin is commonly combined with fatty acids in a 1:3 ratio on a molecular basis to form one molecule of oil for use as a plasticizing additive in paint and adhesive formulations.<sup>84</sup> Due to its hygroscopic nature, glycerol absorbs a large quantity of water. Thus, both gelatin and its added plasticizing agent, glycerin, are biodegradable and retain water in a humid environment. Thus, the susceptibility of this treatment to biological growth, specifically mold, is high. One solution to this problem is to add an antiseptic biocide to the adhesive formulation.<sup>85</sup>

Misting of the treatment area serves two purposes. First it relaxes the surface tension which binds the particles within the detached surface finish together as a rigid mass. The water solution penetrates into the pores of the finish and creates a thin layer of water that

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<sup>83</sup> Ferguson, Ken, Joel Langdon, and Jack Power. "Van Son Printer's Digest: Printing Reference Guide", Van Son Tech Center 3<sup>rd</sup> edition, Van Son Holland Ink Corporation of America, Mineola, NY, p.8. <http://teched.vt.edu/gcc/HTML/VirtualTextbook/PDFs/PrintersDigest.pdf>

<sup>84</sup> Gettens, Rutherford J.; Stout, George L. *Painting Materials : A Short Encyclopedia* Dover Publications Inc., Mineola, NY, 1965.



coats the clay particles within the layer. This thin coating of water is said to activate the clay particles.<sup>86</sup> The shape of clay particles allows them to slide easily over one another making the layer more pliable when pressure is applied.<sup>87</sup> Second, the evaporation rate of isopropyl alcohol is faster than that of water. Thus, a solution of isopropyl alcohol mixed with water theoretically provides a more rapid and more thorough wetting of the pigment particles remaining in the surface finish while also increasing the evaporation rate of this water based solution. The result of better wetting is better coverage of the particles by the adhesive and thus an increase in adhesion. Rapid drying reduces the possibility of overwetting the earthen plaster layers which may deform the stratigraphic sequencing of individual layers.

While resetting the detached finish to its original plane, conservators at the University of Pennsylvania experienced good results when using a cosmetic sponge as a buffer while applying pressure to the detached layer. This evens out the pressure to cover a greater surface area and absorbs any excess solution on the surface of the finish.

### **11.1 Cold Gelatin Treatments**

Cold treatment is the term used to describe application of 5% and 10% gelatin solutions that have been applied in their gelled state. According to the testing conducted for this paper, the gelatin solutions used at Mesa Verde form a firm gel between 20° C. (68° F.) and 25° C. (77° F.) depending on the solution formulation. Cold treatment is

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<sup>85</sup> Matt T. Roberts, Don Etherington and Margaret R. Brown. "Bookbinding and the Conservation of books: A Dictionary of Descriptive Terminology", Stanford University Libraries, 1994.  
<http://palimpsest.stanford.edu/don/don.html>

<sup>86</sup> Brady, Nyle C. and Ray R. Weil. "The Nature and Properties of Soils", 11th Edition, Macmillan Publishing, 1996.

<sup>87</sup> Dix, Linnaea A. "Characterization and analysis of Prehistoric earthen plasters, mortars, and paints from Mug House, Mesa Verde National Park Colorado", University of Pennsylvania Masters Thesis, 1996.



primarily used when the area of detachment is large enough that the adhesive can be applied without the use of a hypodermic syringe. In most cases, cold treatments are applied between a detached layer and its substrate with a microspatula. After the layer has been misted with isopropyl alcohol and allowed to become pliable, a slight pressure is applied to the outermost surface of the detached layer to gently set it back into place.

### **11.2 Warm Gelatin Treatments**

Warm gelatin treatments are applied with a hypodermic syringe at a temperature approximating 36 C. (98° F.). This method of application subjects the adhesive to shear stress while it is passed through the needle. The recommended temperature for warm gelatin treatments is above the melting point for glycerin and above the glass transition temperature for the gelatin. Thus, the mixture is quite viscous and there may be the potential for crosslinking of the gelatin and the glycerin used in this adhesive solution.





## Chapter 12

### ANALYSIS OF ADHESIVE PERFORMANCE

Responsible conservation practice dictates that the conservator understand the material properties of the object to be treated, the properties of materials used to treat it and a consideration for the effects that a treatment will have on the object. Careful assessment of the mechanisms that contribute to the deterioration of the object dictate the treatment method that will be used. Detailed documentation of existing conditions prior to and after treatment provides for the continued monitoring and assessment of specific treatments. This approach has been adopted by the University of Pennsylvania in developing grouts and adhesives for conservation use at archeological sites. It is also the basis for the testing program set forth in this thesis.

Initial testing and analysis to determine the materials characteristics of original surface finishes were determined by visual examination and documentation of in situ plaster finishes during the 2000 and 2001 field season. As a basis for this study, Microscopic cross-sectional analysis was provided by University of Pennsylvania theses and by research by the Getty Conservation Institute. Petrographic analysis of mortar and plaster samples at Mesa Verde was conducted by Mary Griffith<sup>88</sup>, and soils studies were conducted by the Department of Agriculture.<sup>89</sup> Previous surveys of Mesa Verde Surface

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<sup>88</sup> Nordby, Larry and Mary Griffiths. "Cliff Palace Building Materials Classification and Selection", December, 1999.

<sup>89</sup> USDA –NRCS Soil Survey Division. "National SSURGO Database Data Access", January 8, 2001. [http://www.ftw.nrcs.usda.gov/ssur\\_data.html](http://www.ftw.nrcs.usda.gov/ssur_data.html)



Finishes were conducted by Wood Canyon Archeology<sup>90</sup> and Constance Silver<sup>91</sup>. Mechanical testing of the stabilization soils used at Mesa Verde was published by Bob Hartzler in 1996.<sup>92</sup> That same year, Linnaea Dix conducted microscopic analysis of Mesa Verde surface finishes.<sup>93</sup> This study was followed up with the study conducted by Mary Slater in 1999.<sup>94</sup> A third study was conducted by Urs Mueller of the Getty Conservation Institute in 2001.<sup>95</sup> Various treatment methods were laboratory tested in the theses of Maria Isabel Beas,<sup>96</sup> Angelyn Bass-Rivera,<sup>97</sup> and Kechia Fong.<sup>98</sup> Park specific conditions terminology and in-situ testing of treatment methods were conducted by University of Pennsylvania Conservation Laboratory field crews from 1994 to 1997. This research the basis for current conservation practices at Mesa Verde.

The purposes of this study are to assess the properties of materials that are currently being used by the University of Pennsylvania to stabilize original earthen surface finishes and to assess their interaction with the materials that were used to formulate these

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<sup>90</sup> Fetterman, Jerry and Linda Honeycutt, The 1987 Mesa Verde Plaster Recordation Project, Woods Canyon Archaeological Consultants, Inc., Yellow Jacket, CO, 1987.

<sup>91</sup> Silver, Constance.

Analysis and Conservation of Pueblo Architectural Finishes in the American Southwest, Adobe 90, 1990. 1981 Report on the Development of Methods for the Conservation of Pueblo Indian Mural Paintings in the American Southwest, National Park Service, 1982.

The State of Preservation of Pueblo Indian Mural Paintings in the American Southwest, ICCROM, 1980.

<sup>92</sup> Hartzler, Robert. "Acrylic-modified earthen mortar: a program of investigation and laboratory research into acrylic-modified earthen mortar used at three prehistoric Pueblo sites", Conservation Program, Intermountain Cultural Resource Center, Intermountain Field Area, National Park Service, Dept. of the Interior, Santa Fe, NM, 1996.

<sup>93</sup> Dix, Linnaea A. "Characterization and analysis of Prehistoric earthen plasters, mortars, and paints from Mug House, Mesa Verde National Park Colorado", University of Pennsylvania Masters Thesis, 1996.

<sup>94</sup> Slater, Mary E. "Characterization of earthen architectural surface finishes from Kiva Q, Cliff Palace, Mesa Verde National Park Colorado", University of Pennsylvania Masters Thesis, 1999.

<sup>95</sup> Mueller, Urs. "Final Report", The Getty Conservation Institute. Los Angeles, CA, 11/29/2001.

<sup>96</sup> Beas, Maria Isabel G. "Traditional architectural renders on earthen surfaces", University of Pennsylvania Masters Thesis, 1991.

<sup>97</sup> Bass, Angelyn. "Design and Evaluation of Hydraulic Lime Grouts for in Situ Reattachment of Lime Plaster to Earthen Walls", University of Pennsylvania Masters Thesis, 1998.

<sup>98</sup> Fong, Kechia Lee. "Design and Evaluation of Acrylic-Based Grouts for Earthen Plasters", University of Pennsylvania Masters Thesis, 1999.



finishes. First, significant physical properties for plaster formulation were identified. The same was done to identify the material properties of the adhesive formulation currently used in-situ. Next, a sequence of testing was conducted to assess the effects of accelerated weathering of these materials and to assess their interaction with one another.

Factors that were considered in the assessment of original plaster formulations included particle size distribution, particle shape, petrographic and elemental analysis, color, pH, salt content and carbonate content. These same properties, plus an assessment of soil density and plasticity were also noted for locally obtained soils. Further tests to assess the effects of environmentally induced shear stress to strain coefficients were conducted for this study.

To assess the material properties of the University of Pennsylvania's adhesive treatment formulation cure time, cohesive shrinkage, viscosity, reflectance, flexibility, glass transition temperature, vapor transmission and bond strength were each tested. Research was conducted in the fields of biochemistry, photographic conservation, adobe preservation, food science and fine arts painting conservation. Detailed conditions mapping also revealed the short-term effects of environmental weathering. To test the physical interaction between the adhesive and native materials used in plaster manufacture, initial visual observations were combined with measurements of the shear force needed to detach readhered facsimile plasters that were detached from their substrates.

The testing program began with the production of reproduction plaster and wash layers applied to facsimile substrates. Layer thickness, granulometry and porosity of the substrates were based on previous analysis of Mesa Verde earthen renders. Both brick



and sandstone were used as facsimile substrates. Each substrate was tested to determine its percent porosity prior to plaster application. The seven samples were prepared according to the ASTM D 4062-99 Standard Test Method for Leveling of Paints Draw-Down Method, slightly modified to accommodate the thickness of a masonry substrate. Though the method of application was the same, an adjustable draw-down tool was constructed that is able to produce uniform surface finishes on various thickness of substrate. Each of the samples was prepared the same way except for Sample X. All substrates were cut to a thickness in the range of 16-23 mm. with a diamond bit, water lubricated rotary saw. The thickness of plaster layers was gauged according to ASTM D 4138-94(2001)e1 Standard Test Methods for Measurement of Dry Film Thickness of Protective Coating Systems by Destructive Means. The underlying coat of reddish brown plaster was formulated with a sand:silt/clay ratio of 65:35 and was applied at a thickness within the range of 1.2-1.3 mm. The top layer of wash consisted of a 35:65 sand:silt/clay ratio applied at a thickness of 0.4-0.5 mm. Sample X was a thin slice of sandstone that was coated with four layers of plaster. The first layer was a mixture of the reddish brown (SY-3) soil mixed with the same ratio as above. The second layer was made from the same mixture of very pale brown (SY-2) soil and was followed with a layer of white soil (SY-1). The white soil consisted of 50% very pale brown sand, 30% very pale brown silt and 20% white silt.

The intention of these tests was to try different variations of temperature and moisture cycling to determine a method most likely to produce the blistered and delaminated condition that is common at Mesa Verde. These experiments were terminated once the samples showed a sign of either blistering or cracking.





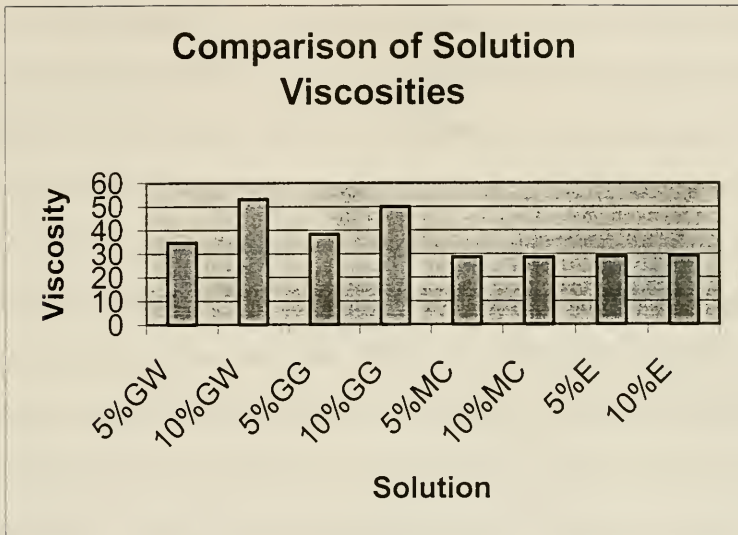
### **12.1 Viscosity Test**

Samples of each of eight solutions were subjected to three tests with appropriate Zahn viscosity cups. Tests were conducted according to ASTM D 4212-99 Standard Test Method for Viscosity by Dip-Type Viscosity Cups. The solutions were heated and cooled numerous times throughout the experiment in order to obtain exact temperatures for testing. However, this should not induce a high margin of error because each solution was subjected to the same procedure. Viscosity tests of gelatin based solutions at colder temperatures were attempted with Zahn cups #3 and #4. However, the gelatin did not solidify uniformly throughout the solution at a consistent rate. Thus, interruptions in flow of the solution affected the timing of these tests producing inconsistent results. Since the viscosity of gelatin-based solutions vary with slight temperature changes between 20-25 degrees Celsius, additional tests were conducted at higher temperatures for the gelatin-based solutions. Ambient temperature during the course of this experiment was 21°C with 22% relative humidity.



TABLE 23

Viscosity of Treatment Solutions



KEY

5%GW = 5% Gelatin in water

10%GW = 10% Gelatin in water

5%GG = 5% Gelatin with 10% Glycerin in water

10%GG = 10% Gelatin with 10% Glycerin in water

5%MC = 5% Roplex MC-1834 in water

10%MC = 10% Roplex MC-1834 in water

5%E = 5% Roplex E-330 in water

10%E = 5% Roplex E-330 in water



The acrylic emulsions were less viscous than the gelatin solutions, which explains why the acrylic solutions were more likely to cause staining when applied in the field. Staining was likely caused by the more thorough coverage of soil particles and reverse migration of the suspended solids. The viscosity of acrylic emulsions tested was equivalent to the viscosity of de-ionized water without the added adhesive component. Viscosity of the gelatin solutions varied according to the percentage of gelatin within the solution. Other determinants of viscosity of gelatin solutions that were not specifically tested include temperature, molecular weight and hydrogen-ion concentration.<sup>99</sup> The molecular weight of gelatin varies from 20,000 to 250,000<sup>100</sup> and has a direct impact on the adhesive bond and gel strength of a gelatin solution. "The lower the mean molecular weight (MW) of a gelatin the lower the gel strength and viscosity of its solution, however it has been shown that the collagen alpha-chain is the main contributor of gel strength and that higher molecular-weight components make a relatively low contribution to gel strength but a high contribution to viscosity... In general one can say that the lower the mean molecular weight (MW) of a gelatin the lower the gel strength and viscosity of its solution."<sup>101</sup> Since the molecular weight of gelatin can vary greatly depending on the raw materials used and method of manufacture, viscosity is an important factor in determining the adhesive capability of a gelatin solution. Thus, a major factor for

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<sup>99</sup> Cole, Bernard. "Gelatine Food Science", University of Pretoria, <http://www.gelatin.co.za/gltln1.html>

<sup>100</sup> Matt T. Roberts, Don Etherington and Margaret R. Brown. "Bookbinding and the Conservation of books: A Dictionary of Descriptive Terminology", Stanford University Libraries, 1994. <http://palimpsest.stanford.edu/don/don.html>

<sup>101</sup> Cole, Bernard. "Gelatine Food Science", University of Pretoria, <http://www.gelatin.co.za/gltln1.html>



assessing commercial grades of gelatin is the calculation of viscosity by a mathematical model to calculate Bloom.

Most gelatin-based films require a low viscosity to enable wetting and coverage of the adherent surfaces. Addition of a gelatin plasticizing agent lowered the viscosity of a 10% Gelatin solution. Based on the results of this test, the higher the concentration of gelatin within the solution, the higher the temperature needed to obtain equivalent amounts of viscosity.

### **12.2 Set Time**

This test was loosely based on D 2471-99 Standard Test Method for Gel Time and Peak Exothermic Temperature of Reacting Thermosetting Resins and D 4640-86(2001) Standard Test Method for Determining Stroke Cure Time of Thermosetting Phenol-Formaldehyde Resins and D 4473-01 Standard Test Method for Plastics: Dynamic Mechanical Properties: Cure Behavior and D 3056-00 Standard Test Method for Gel Time of Solventless Varnishes. In addition, a glazed ceramic spot test plate was filled with a 0.05 cc. of each solution. The ambient environment was 21° Celsius at 22% relative humidity. The tray was placed in the vent hood and samples were monitored on an hourly basis. The following solutions were tested: 5% Rhoplex E-330 in water (5%E), 10% Rhoplex E-330 in water (10%E), 5% Rhoplex MC-1834 in water (5%MC), 10% Rhoplex MC-1834 in water (10%MC), 5% Gelatin in water (5GW), 10% Gelatin in water (10%GW), 5% Gelatin with 10% Glycerin in water (5%GG), 10% Gelatin with 10% Glycerin in water (10%GG).





#### Observations at 1 hour:

All gelatin solutions were in solid/gel form containing approximately the same volume as they did in liquid form. The acrylic solutions were approximately 75% evaporated as determined through visual examination. A cracked film had formed on the surface of 10%MC and an extremely thin, uncracked film had formed on the surface of the 10%E.

#### Observations at 2 hours:

Gelatin solutions were still in solid/gel form and only 25% of the original volume had evaporated. All acrylics had more than 75% evaporated and a surface film has formed on each sample.

#### Observations at 3 hours:

More than 75% of all solutions had evaporated. All acrylic solutions were white, rigid films adhering to the surface of the ceramic test plate. Gelatin solutions in water formed thin, rigid films that cohesively pulled up from the bottom of the test plate forming their characteristic air bubble beneath them. Gelatin in water solution films were of a transparent, slightly yellow coloration. Gelatin and glycerin solutions adhered to the surface of the test plate, were transparent and remained tacky to the touch.

#### Observations at 4 hours:

Same as observations at 3 hours except that the acrylic films changed to a clear coloring. One method for establishing the grade of gelatin used in a solution is the determination of Gel/Bloom Strength. This is determined as follows by measuring the force needed to compress a specified amount of gelatin while in a gelatinous state. Laboratory methods for establishing the Bloom Strength are outlined in British Standard



757 of 1975 and in the Standard Methods for sampling and testing gelatin, published by the GMIA.<sup>102</sup>

### **12.2.1 Glass Transition Temperature**

Previous studies of the glass transition temperature of gelatin determined their liquid limit to be 35° Celsius and solid limit to be at 10° Celsius. The term glass transition point is commonly used to describe the point where a film converts from a liquid to a solid. For gelatin solutions, the transition from a liquid to a solid is dictated by two factors. The initial transition from liquid state to solid for this treatment is dictated by temperature while the final transition is the result of solvent loss through evaporation. Testing was conducted to determine the temperature that liquid gelatin formulations form a gelatinous mass. This gel point was recorded for each gelatin based solution used in this study. The test was conducted at an ambient temperature of 66 degrees Celsius at 42% relative humidity. This test was conducted by warming 500 ml of each solution to attain a fully liquefied state. The solutions were then left to cool. Solutions were stirred every fifteen minutes with a glass stirring rod. Temperatures were recorded at the point when gelatinous solutions became solid enough to support the glass stirring rod in a vertical position within the solution. This test produced the following results.

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<sup>102</sup> Cole, Bernard. "Gelatine Food Science", University of Pretoria, <http://www.gelatin.co.za/gltm1.html>



### Liquid to Gel/Solid Transition Point

- 5% Gelatin in water = 21 degrees Celsius
- 10% Gelatin in water = 23 degrees Celsius
- 5% Gelatin and 10% Glycerin in water = 24 degrees Celsius
- 10% Gelatin and 10% Glycerin in water = 24 degrees Celsius

### 12.3 Reflectance Test

Observations were made according to ASTM D 4449-90 Standard Test Method for Visual Evaluation of Gloss Differences Between Surfaces of Similar Appearance with Lamp B as specified in this test. Reflectance was most noticeable with the lamp positions at a 90-degree angle to the specimens and located 10 inches above them. Observations were made at a 45-degree angle to the specimens. Observations were recorded on a scale of 0-5 with 5 having the most reflectance and 0 having no visible reflectance.

Microscopic observations were made one week after treatment. Samples 1-3 were tested with solutions heated to a temperature of 65 degrees Celsius (see table 24).



TABLE 24

## Test Data for Assessing Reflectance

Formulation	Sample #1 - White Wash		Sample #2 - Brown Plaster	Sample #3 - Brown Wash	
	Dry Substrate	Pre-wet Substrate	Dry Substrate	Dry Substrate	Pre-wet Substrate
1%MC	0	0	0	0	0
3%MC	0	1	1	1	0
5%MC	2	3	2	3	1
10%MC	5	4	3	4	3
1%E	0	0	0	0	0
3%E	2	2	1	1	0
5%E	3	3	3	3	2
10%E	5	4	4	4	3

Both the 5% and 10% mixtures of gelatin without glycerin on white plaster shrank upon cure when applied to dry calcium carbonate plaster. Thus, their cohesive strength was stronger than that of the wash resulting in cracks and flaking where the solution was in contact with the wash. The reddish brown surface finishes exhibited less reflectance than the white wash did. The reddish brown plaster, formulated with a larger amount of sand sized particles exhibited less reflectance than both of the wash samples. As predicted, a higher the percentage of acrylic produces an increase in reflectance (see table 25 and 26).





TABLE 25

Reflectance of Acrylics when Applied to Earthen Surface Finishes

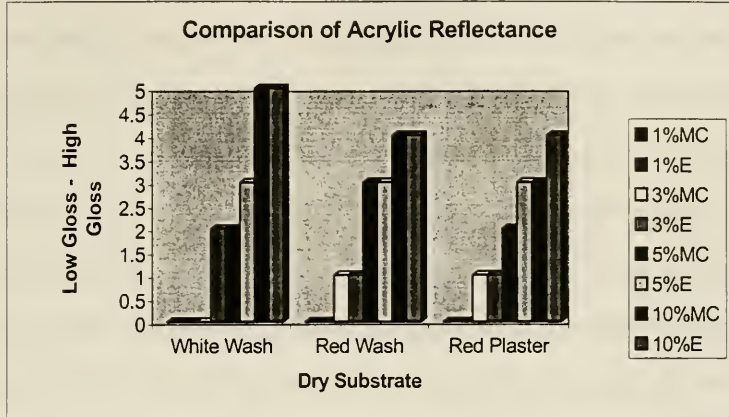
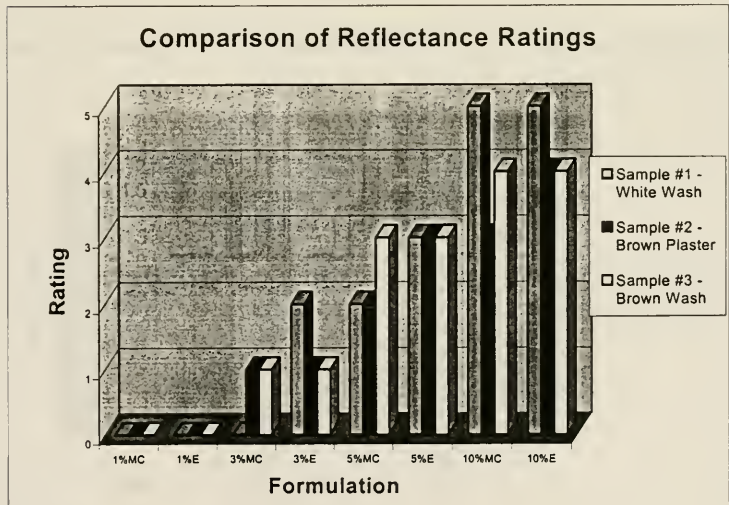


TABLE 26

Comparison of Reflectance Ratings for Adhesive Formulations

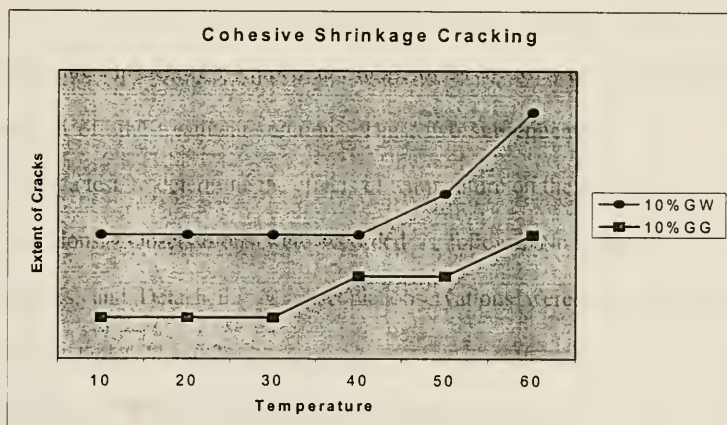




During the gloss test, it was noted that shrinkage cracks and detachment occurred where the treatment contacted the surface finish. In the field, solutions are used within the temperature range from 10°-40° Celsius. Applications recorded as “hot treated” are heated on site to a temperature of approximately 35° - 37° Celsius. “Cold treated” applications are injected at a temperature range of 21° - 26° Celsius depending on the concentration of the treatment solution. Thus, two subsequent tests were conducted. The first was a test to determine the effects of temperature on the cohesive contraction of gelatin solutions. Observations were recorded as follows: No Cracks, Micro-Cracks, Macro-Cracks, and Detached. Microscopic observations were made one week after treatment (see tables 27 and 28).

**TABLE 27**

**Size of Cracks formed by Unrestrained Cohesive Shrinkage of Adhesives**





**TABLE 28**  
**Shrinkage Observations for White Wash Sample #4**

<b>Temperature</b>	<b>10%GG</b>	<b>10%GW</b>
20 Degrees Celsius	Micro-Cracks	Macro-Cracks
30 Degrees Celsius	Micro-Cracks	Macro-Cracks
40 Degrees Celsius	Micro-Cracks	Macro-Cracks
50 Degrees Celsius	Macro-Cracks	Micro-Cracks
60 Degrees Celsius	Macro-Cracks	Macro-Cracks
70 Degrees Celsius	Detached	Detached

#### **12.4 Shrinkage of Solutions**

A second test was conducted to determine any difference in cohesive shrinkage cracking between solutions of gelatin with glycerin (as an added plasticizer) and solutions of gelatin without an added plasticizer. Ambient conditions during treatment was 22°C and there was 30% relative humidity. Temperatures selected for this test were within the range where these solutions begin to gel. Observations were recorded 24 hours after treatment at 23°C with 20% relative humidity and at one week after treatment. They were recorded according to the following scale: No Cracks, Micro-Cracks, Macro-Cracks and Detached. Microscopic observations were made at 30X magnification.

The treatment that is currently in use at Mesa Verde is formulated with a protein binder that consists of reagent grade gelatin, often mixed with the alcohol glycerin. This formulation is common. As Thorton explains, "It has long been understood that glycerin added to animal glue will give it greater toughness and elasticity when dry. If glycerin is added in sufficient quantities (a 1:1 dry glue weight to plasticizer volume mixture ), it



will prevent the glue from hardening altogether, the result being a rubber-like substance.”<sup>103</sup> The effectiveness of this plasticizing additive was exhibited in the materials testing conducted for this study. Gelatin is a polymer that may physically bond with glycerin when combined into solution. However, the two components of this solution do not crosslink on a chemical level at room temperature.<sup>104</sup> When applied to the nonabsorbent substrate of a spot-test plate, the gelatin and glycerin remained suspended within the solution. However, it may be possible for these physical bonds to be broken. The effectiveness of glycerin as a plasticizing agent is dependant upon the strength and durability of these physical bonds.

Therefore, this test was run two times. The first attempt at this test used solutions that had been heated, cooled and reheated numerous times before being used. The results of that initial test were inconsistent with the results found when the same solutions were applied to a glazed ceramic substrate. During this initial test, both plasticized and unplasticized gelatin solutions exhibited cracking at the junction between adhesive and substrate. One possible cause is that when plasticized solutions are applied to a desiccated substrate, the glycerin absorbs into the substrate at a more rapid rate than the gelatin resulting in the same results for both solutions. Another explanation may be that the warming and cooling of this solution induced evaporation thus changing the percentage of each component within the solution. Since human error could not be ruled out, the test was conducted again with newly mixed solutions.

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<sup>103</sup> Thornton, Jonathan. “A Brief History and Review of the Early Practice and Materials of Gap-Filling in the West”, *Journal of American Institute for Conservation* 1998, Volume 37, Number 1, Article 2, p. 3-22.

<sup>104</sup> Personal Communication with Lee Jhong, Rheological Chemist and speaker at the 31<sup>st</sup> International Symposium of Archeometry, 3/5/2002.





The results of this second test consistently revealed that the cohesive strength of a gelatin solution that does not contain a plasticizing additive such as glycerin is much greater than the cohesive strength of the earthen surface finishes. This results in cracks forming where the forces of internal contraction within a gelatin formulation are stronger than the cohesive forces within the finish layer. Since the cohesion within a finish layer is stronger than the adhesion between one layer and the next, there is a plane of weakness between each layer of surface finish. When subjected to external stresses, the resulting fracture often occurs at this plane. The differential rate of contraction for unplasticized gelatin formulations and surface finishes resulted in crack formation at the boundary of the treated area and interfacially along this plane of weakness. The 10% gelatin solution in water contracted so much as it set that, within a week, most of the treated areas where solution was originally applied to a sound substrate of surface finish had detached completely and exhibited finish loss. This effect makes it clear that use of an unplasticized gelatin solution is inappropriate in conditions where the gelatin is subjected to fluctuations in temperature and relative humidity. In fact, without the addition of a plasticizer in gelatin solutions, this adhesive could cause deterioration of surface finishes.

This consideration should not be lost when determining the percentage of plasticizer to gelatin within a solution. Both gelatin and glycerin retain moisture within the structure. Unlike colorless gelatin that is precipitated from salts or alcohol, gelatin produced by changes in pH or temperature has a yellow cast to it when solid. This yellow gelatin is documented to retain 15-18% water in solid, dry form, making it more flexible.<sup>105</sup> Over

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<sup>105</sup> Roberts, Matt T. and Don Etherington. "Bookbinding and the Conservation of books: A Dictionary of Descriptive Terminology", Stanford University, 2001. <http://palimpsest.stanford.edu/don/don.html>



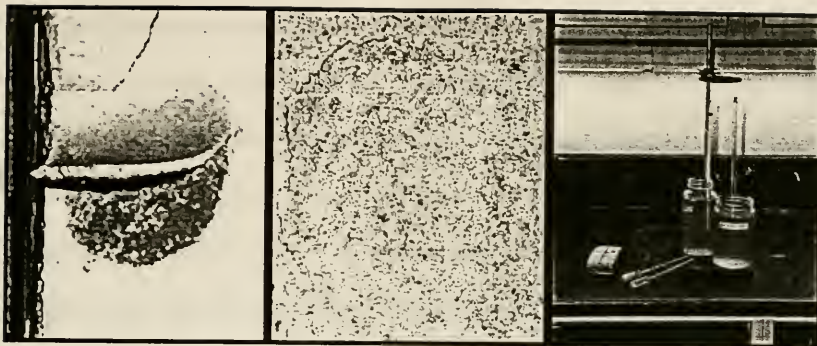
time, the amount of water that gelatin is able to retain within its structure may change, making this adhesive more brittle. The effects of gelatin embrittlement are documented in numerous studies for the conservation of historical oil paintings. The pH of the gelatin also affects its hardness, which may be a factor in the embrittlement of gelatin used for painting and bookbinding. "Hardness of the gel is a maximum at pH 8.5 and decreases on either side of this pH."<sup>106</sup> Gelatin and glycerin are not chemically bonded when suspended within this adhesive solution. They are physically attracted to one another, but with time, the glycerin may be absorbed into the substrate leaving the gelatin unplasticized. This is one instance where the acrylic solutions are more practical because they are chemically bound, otherwise known as internally plasticized. Research is currently being conducted to test the crosslinking of gelatin solutions with acrylics.

Due to surface finish detachment and cracking observed while testing for reflectance and cohesive shrinkage, the penetration of gelatin solutions could be observed. Amounts of solution were applied to the surfaces of reproductions made up of a 4-mm-thick plaster overlaid with a 1-mm wash layer. The solution never penetrated into the plaster layer. Thus, these gelatin solutions did not penetrate more than 1 mm into the reproduction surface finishes. This was evidenced because as the gelatin solutions cured, they contracted and lifted up from their plaster substrate. When applied to plaster without a wash, gelatin solutions did not lift from the surface but microscopic cracks did occur. For more data regarding this test refer to Appendix F.

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<sup>106</sup> Roberts, Matt T. and Don Etherington. "Bookbinding and the Conservation of books: A Dictionary of Descriptive Terminology", Stanford University, 2001. <http://palimpsest.stanford.edu/don/don.html>





Figures 18 and 19. Shrinkage cracks formed  
by unplasticized gelatin

Figure 20. Testing apparatus  
for Shrinkage and Reflectance

#### 12.4.1 First Test for the Relative Shrinkage of Solutions

A sample of 3 cc. solution of each material was placed in a black glazed ceramic spot test plate. The acrylics were applied at a temperature of 23 degrees Celsius and the gelatin solutions were applied at 35 degrees Celsius to ensure fluidity. The atmospheric temperature at the time of application was 23 degrees Celsius and the relative humidity was 35%. Observations were made 96 hours later at an atmospheric temperature of 21 degrees Celsius and 41% relative humidity. Observations were based on tactile, visual and volumetric comparison. Visual comparisons were made with the naked eye and microscopically. Multiple tests of each of the following were made for relative comparison.

#### Observations:

The gelatin in water solutions formed a rigid, hard, cohesive film that adhered to itself better than it adhered to the glazed ceramic substrate. Due to the cohesive nature of this solution, an air bubble formed on the underside of the film in every sample. The size



of air bubbles formed below films of the 10% solution were visually approximated at twice the size of that formed with the 5% solution. The cohesive nature of the gelatin films effectively sealed the solution underneath from the air. Thus, even after 96 hours, a portion of the 5% solution was not fully cured, but instead was trapped in the bubble underneath a rigid film. Only as the gelatin contracted cohesively, did the sides of this film pull away from the walls of the cup exposing the solution beneath it to the air and thus allowing it to cure.

Gelatin with glycerin used as an additive formed a less rigid but just as cohesive film. No air bubble was trapped beneath the film. Furthermore, the film remained tacky to the touch 96 hours after the solution was applied.

Both acrylic resins formed hard films that were less cohesive than the gelatin solutions. All acrylic films cracked as they set and no air bubbles were formed. This reaction can be explained by applying the model of evaporative drying used to explain the cracked appearance of some latex paints. "As the film dries from the surface down, a fixed film area is then subject to contraction in the z-plane, thereby producing stress in the x-y plane. If polymer elasticity is insufficient, then the stress can be overcome by slippage between the coalesced layer and the fluid beneath giving rise to the 'mud-cracked' surface effect."<sup>107</sup>

#### **12.4.2 Second Test for the Relative Shrinkage of Solutions**

An additional test was conducted by putting 9 cc. of each adhesive into a 50 ml. graduated cup. The shrinkage of each sample was rated from 0 – 10 with 10 denoting the

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<sup>107</sup> Roberts, Matt T. and Don Etherington. "Bookbinding and the Conservation of books: A Dictionary of Descriptive Terminology", Stanford University, 2001. <http://palimpsest.stanford.edu/don/don.html>





most shrinkage and 0 denoting the least amount of shrinkage once cured. Volumetric data was obtained by recording the number of cc. needed to fill the cup, atop of the dried film, to achieve the same level of fullness. Observations of volume loss were made 336 hours later at an atmospheric temperature of 20° Celsius and 17% relative humidity.

**TABLE 29**  
**Test Data for Volumetric Shrinkage Test**

<b>Sample Solution</b>	<b>Volume Lost (cc. of solution)</b>
5% Gelatin in Water	8 cc.
10% Gelatin in Water	8 cc.
5% Gelatin with 10% Glycerin in Water	8 cc.
10% Gelatin with 10% Glycerin in Water	8 cc.
5% E-300 in Water	7 cc.
10% E-330 in Water	7 cc.
5% MC-1834 in Water	7 cc.
10% MC-1834 in Water	7.5 cc

Studies have shown that swelled gelatin is able to retain ten times its dry weight in moisture when in gel form.<sup>108</sup> Yet in solid form, there was little difference between the gelatin and acrylic-based solutions. Thus, the material properties of gelatin solutions are equivalent to that of acrylic solutions when not exposed to great fluctuations in temperature and relative humidity. Unfortunately, climate control is not possible in the field.



## 12.5 Expansion, Contraction and Flexibility of Adhesives

To provide a relative comparison of how each formulation would react to the wetting and drying cycles it might be exposed to in the field, each of the cured solutions used in the first test for relative shrinkage were rewetted with 2.5 cc of water retained at a temperature of 10° Celsius. The solutions were left to soak in this water bath for 24 hours, then observed. Once dry, they were again rewetted one more time. Both visual and tactile observations were made when the solutions were fully dry. The following solutions were tested: 10% gelatin in water, 10% gelatin with 10% glycerin in water, 10% MC-1834 in water, 10% E-330 in water (see figures 21).

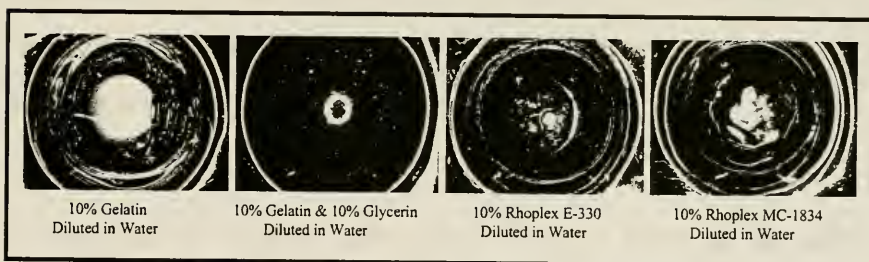


Figure 21. Spot Test Plates Containing Cured Adhesives

### 12.5.1 Acrylic solutions

While wet, the acrylic solutions resumed their white color but did not return to solution (see figure 22). Upon drying, the adhesive films exhibited the same transparent color and the same “mud-cracking” that occurred during the initial cure of these films. It is notable that the acrylic films were able to be removed from the ceramic spot plates

<sup>108</sup> Cassar, JoAnn and Roberta de Angelis. “Glossary. Materials used in 19th and 20th century Plaster Architecture. Plaster Architecture Essay, Culture 2000 Programme of the European Union. <http://www.plasterarc.net/essay/essay/CassarG.html>



only after soaking and vigorous scrubbing. Thus, removal of acrylic adhesives with a solution consisting solely of water would not be reasonable in the context of earthen surface finishes.

#### **12.5.2 Unplasticized gelatin solutions**

This was the only solution that did not remain closely adhered to itself throughout the duration of this experiment. Cohesive contraction of this gelatin solution during its initial cure caused it to pull away from the ceramic dish. Each successive wetting and drying episode caused the unplasticized gelatin film to exacerbate this affect by pulling up from the center of the cupped portion of the ceramic spot plate. By the end of the second wetting and drying period, the unplasticized gelatin solution had become fully detached from the glazed ceramic substrate. This affect was not observed for any of the other solution types.

#### **12.5.3 Plasticized gelatin solutions**

While wet, the plasticized gelatin solutions took on a yellow cast and swelled slightly but did not return to gel form at room temperature. The solution remained tacky to the touch after two wetting and drying cycles, and remained closely adhered to the ceramic spot plate.



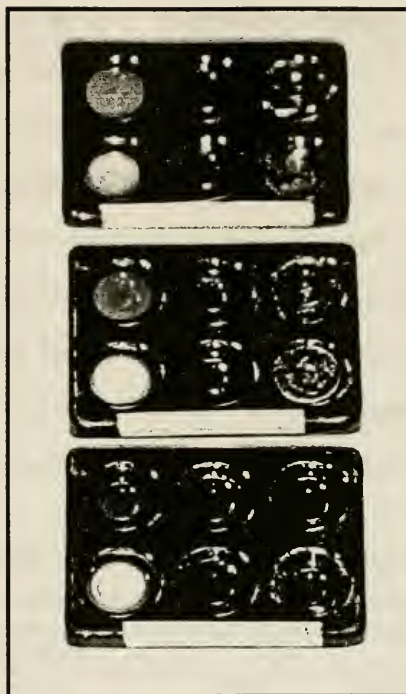


Figure 22. Spot Test Plates Containing Rewetted Adhesives

#### 12.4.4 Elasticity

Elasticity, commonly recorded as E-modulus in paint and adhesive formularies, has a highly influential affect on the performance of such films. As stress builds up interfacially between layers due to fluctuations in temperature and relative humidity, this stress causes strains that result in cracking and adhesion loss. If the adhesive is less rigid and more flexible than the adherents, it may serve as a buffer to absorb these stresses.<sup>109</sup> So long as the adhesive does not impose new stresses on the object being conserved, as

<sup>109</sup> Berger, Gustav A. and William H. Russell. "Conservation of Paintings : Research and Innovations", Archetype Publications: London, 2000.





the adhesive reacts to these same environmental fluctuations, elasticity is a most favorable quality when selecting an adhesive. As gelatin ages, it becomes more completely crosslinked resulting in a more rigid and brittle bond. This brittleness results in a loss of bond strength for the original unplasticized gelatin adhesive and a reduction in bond strength for future treatments as well. Materials testing published in the Conservation of Paintings tested the bond strength of unplasticized gelatin solutions to that of gelatin solutions that were plasticized with honey.<sup>110</sup> The results of this experiment exhibited no difference in bond strength (see table 30). However, the plasticizing effects of glycerin was not tested in this experiment.

**TABLE 30**  
**Materials testing to Determine the Bond Strength for Retreatment of Adhesives**

Peel test group (d): peel strength of different adhesives on paint films impregnated with gelatin and microcrystalline wax (ASTM D 903-49)			
Composition of adhesive	Cleaned, unimpregnated control	Paint film impregnated with:	
		gelatin in water 1:16	microcrystalline wax (Victory White)
Fish glue + honey in water 1:1:10	failure	failure	failure
Gelatin in water 1:10	failure	failure	failure
Rabbitskin glue in water 1:10	failure	failure	failure
Gelatin + honey in water 1:1:10	failure	failure	failure
Rabbistkin glue + honey in water 1:1:10	failure	failure	failure
Elvacet 1454 in water 1:2	400	failure	failure
Thermogrip in benzine 1:3	750	20	30
Beeswax, multiwax, Dammar, Venice turpentine 6:6:4:1	135	80	50
Microcrystalline wax (Victory White)	100	100	50
All values in grams per 12.7 mm at the cross-head speed of 1 mm/min.			
Failure - spontaneous delamination prior to test.			

Acrylics are generally considered to be internally plasticized. An internally plasticized adhesive is defined as "An adhesive which has had the plasticizing agent

<sup>110</sup> Berger, Gustav A. and William H. Russell. "Conservation of Paintings : Research and Innovations",



introduced as part of the adhesive molecule during the manufacturing process, as contrasted with an adhesive to which the plasticizer is added by the user. The adhesive is copolymerized with the plasticizer to form the internally plasticized adhesive.”<sup>111</sup>

By the definition noted above, the gelatin used in these formulations is unplasticized until the user adds an external plasticizing agent such as glycerin. In theory, the adhesive solution of 10% gelatin in water would be less elastic than the 10% solution that is combined with an equal percentage (weight to volume) of the plasticizing agent glycerin. This was proved a valid assertion in the tests conducted for this study.

One advantage to using an internally plasticized adhesive is that adhesive agent and plasticizing agent will not separate without human intervention so long as they are stored within the normal range of environmental fluctuation. However, internally plasticized adhesives are often less reversible and the crosslinking of some adhesive formulation can eventually cause the solution to become embrittled. Crosslinking within unplasticized gelatin solutions can cause the embrittlement of such films thus inducing the damage seen in cracked oil paintings that have been sized with gelatin. Physical bonding of gelatin to glycerin is possible, but chemical crosslinking is not possible at room temperature.<sup>112</sup>

Several methods were devised to measure the elasticity of each 10% solution. The simplest test for elasticity was simple visual and tactile observations made during the course of this experiment. At the end of this experiment, the unplasticized gelatin films

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Archetype Publications: London, 2000.

<sup>111</sup> Matt T. Roberts and Don Etherington. Bookbinding and the Conservation of books: A Dictionary of Descriptive Terminology <http://palimpsest.stanford.edu/don/don.html>

<sup>112</sup> Personal Communication with Lee Jong, Rheological Chemist and speaker at the 31<sup>st</sup> International Symposium of Archeometry, 3/5/2002.



were already detached, and the plasticized gelatin films were carefully removed from their substrate with a microspatula. The unplasticized gelatin film was hard and rigid while the plasticized gelatin film was still elastic and pliable.

### **12.6 Adhesive bond strength**

After five cycles of accelerated weathering, 27 samples were treated with an adhesive solution and mechanically tested to determine the relative bond strength of such solutions. All samples were treated for both substrate detachment and interlayer detachment.

Three reddish brown samples were treated with each of the following solutions:

- (1) water
- (2) 5% gelatin in water
- (3) 10% gelatin in water
- (4) 5% gelatin combined with 10% glycerin in water
- (5) 10% gelatin combined with 10% glycerin in water
- (6) 5% Rhoplex MC-1834 acrylic in water
- (7) 10% Rhoplex MC-1834 acrylic in water
- (8) 5% Rhoplex E-330 acrylic in water
- (9) 10% Rhoplex E-330 acrylic in water.



Three samples of white caliche finishes were also treated with these solutions:

- (1) water
- (2) 5% gelatin combined with 10% glycerin in water
- (3) 10% gelatin combined with 10% glycerin in water
- (4) 10% gelatin in water
- (5) 10% Rhoplex MC-1834 acrylic in water
- (6) 10% Rhoplex E-330 acrylic in water.

A single seven-day cycle of weathering was conducted on all samples after treatment. Due to the already severely deteriorated state of these samples, the procedure for this weathering cycle was limited to fluctuations in the level of moisture applied to one half of the sample. Misting of finish surfaces was conducted hourly for the first five hours. Then samples were given a two-hour period to dry before being misted hourly for the next five hours. At the end of this seven-day cycle, all samples were examined and photographed.

Since full substrate detachment was consistent for all samples prior to treatment, mechanical testing was conducted on treated and weathered samples to determine the relative bond strength of each adhesive at this juncture. Two tests were developed to measure the amount of shear force required to break the bond between substrate and plaster finish. In an effort to devise an inexpensive testing method that can be done in the field, a hand-held force meter was retrofitted with an attachment to disperse the force needed to detach the finishes from their substrate (figure 24 and 24). This method of detachment was effective in providing relative measurements for bond strength of 5%





solutions, except that the force needed to break a bond created with 10% solutions was beyond this tool's maximum capacity. For this, an Instron model 1331 was utilized. Both testing setups are illustrated in figures 25 and 26.

One notable difference between the two tests is that the Instron graphs minute fluctuations in adhesive bond strength such as cracking of the material prior to full detachment. In some cases, the finishes detached in a single, intact layer. This type of detachment was attributed to the use of an adhesive that was weaker than the cohesive strength of the finishes. Other samples crumbled and cracked rather than fully detached from their substrate. This result was interpreted as evidence that the adhesive was stronger than the original finish materials.

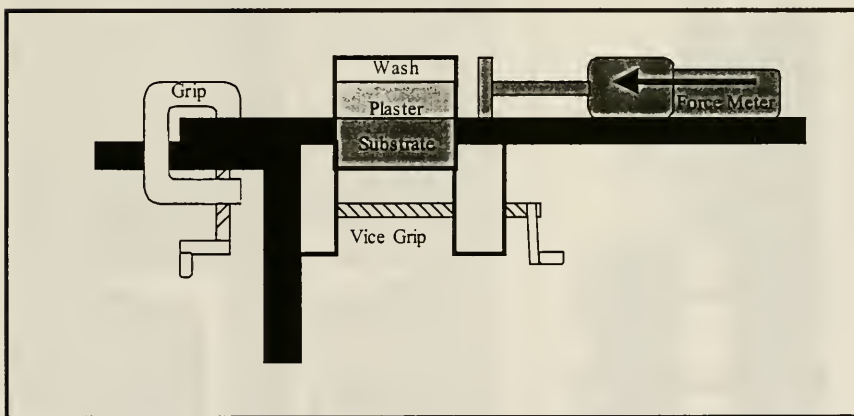


Figure 23. Illustration of the Testing Set up for applying force to treated Samples



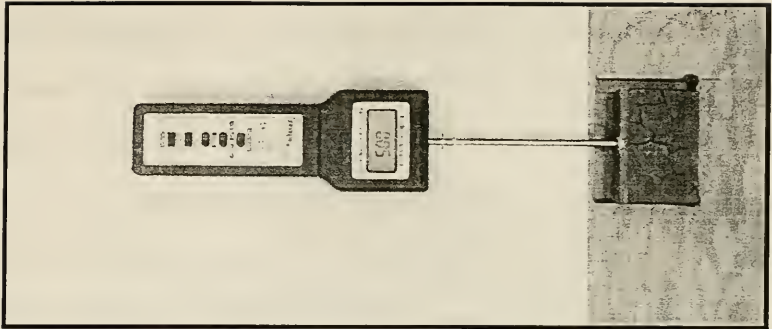


Figure 24. Adhesive bond strength testing conducted with the hand-held force meter

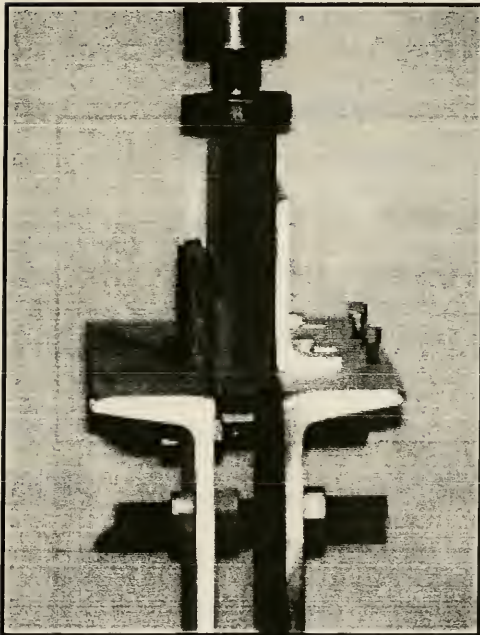


Figure 25. Adhesive bond strength testing  
with the Instron model 1331

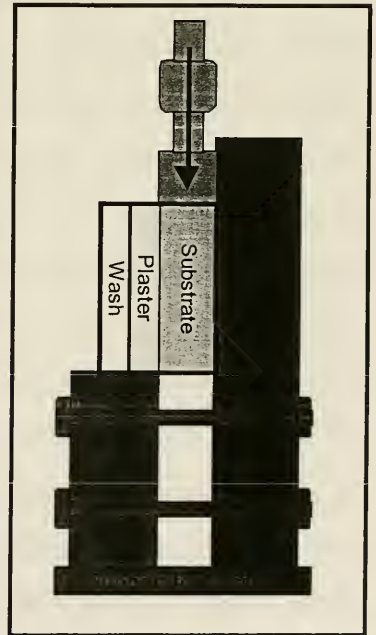


Figure 26. Illustration of Instron  
model 1331 testing method



A comparison of these results shows that the Rhoplex MC-1834 solutions have the highest bond strength. In fact, the mean value of force needed to break the adhesive bonds formed with a solution of 10% gelatin and 10% glycerin in water is equivalent to that needed to break a bond formed with a solution of 5% Rhoplex MC-1834 in water (see table 31). The difference between a 5% and 10% solution of Rhoplex MC-1834 was more than double while the same increase in solids content for Rhoplex E-330 resulted in a nominal increase in bond strength (see table 32).

Visual comparisons of fractures induced by this indicated a tendency for both 10% gelatin solutions and the 10% MC-1834 acrylic solution to suffer fractures within the plaster and wash layers before the plaster layer fully separated from its substrate. This indicates that either the adhesive is stronger than the adherents that it bonds or that the sample was so weathered that it fractured along flaws that already existed within the reproduced surface finishes. If this first assumption is correct, the cohesive strength of weathered reproduction plaster layers lies just under 300 pounds of force. Furthermore, the crumbling observed to occur with samples that were treated with a solution of 10% gelatin in water crumbled differently from those of the other samples (see figure 113). While the unplasticized 10% gelatin solution crumbled into small fragments, the plasticized gelatin solution and MC-1834 acrylic solution each retained their original form despite the new cracks and delamination that occurred. This is significant because it may indicate that earthen plasters that are treated with the gelatin with glycerin may be more resistant to losses caused by physical impact than surfaces treated with an unplasticized gelatin solution.





Figure 27. Sample exhibiting fractures that indicate a stronger adhesive bond strength than the cohesive strength of the surface finishes

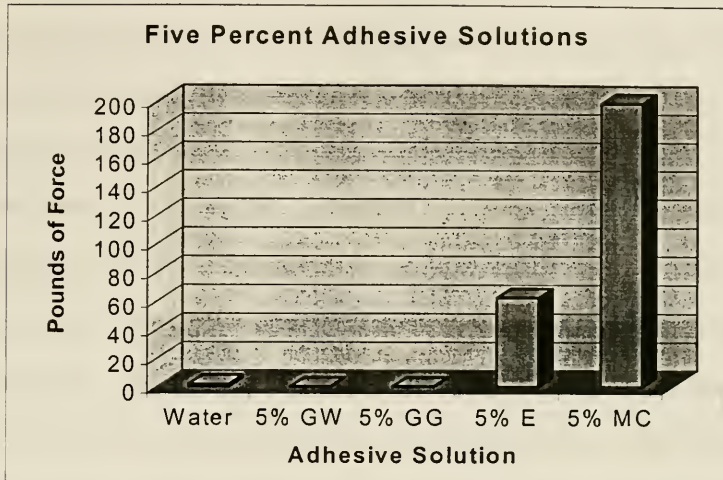
Further deterioration occurs because the use of less binder allows more moisture to penetrate it. The ratio of particle content to binding medium determines how well the particles are coated with binding medium. An excess of binding medium can serve as a sealant by filling the pores of both surface finishes and substrate. Since wash layers are formulated with a greater quantity of clay and silt sized particles, they are less porous than paints with larger particle sizes. When water enters the masonry substrate by means of rain, percolation or capillary action it is retained at the plaster-to-wash interface because the plaster formulation allows for more moisture permeability than the wash does. When the wash is not chemically formed through crystallization, these small binding particles are free to expand with the retained water. This expansion increases the rate of isolated detachment between plaster and wash that is characterized by blistering.





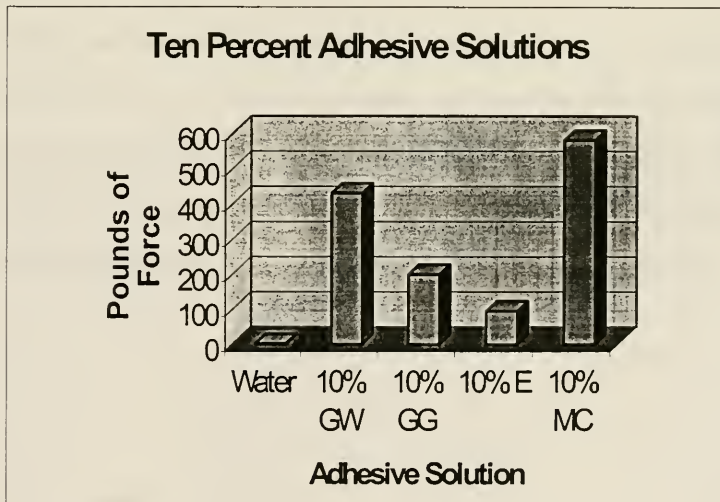
**TABLE 31**

**Comparison of Bond Strength for 5%Gelatin to 5%Acrylic Formulations**



**TABLE 32**

**Comparison of Bond Strength for 10%Gelatin to 10%Acrylic Formulations**





### **12.7 Vapor Transmission Test**

Procedures for this experiment are outlined in ASTM D 1653-93 Standard Test Methods for Water Vapor Transmission of Organic Coatings, Test Method B. Eighteen samples of 60:40 sand:silt/clay ratio were cast to form circular discs of a 1.5 cm. thickness and a diameter of 8 cm. Samples disks were allowed to sit in 30%RH for 72 hours after air-drying. Coatings were applied with a camel-hair brush in two successive coatings applied at 90° angles from one another. After another 72 hours of drying in a desiccator maintained at 20° Celsius and 30% relative humidity. Samples were sealed in plastic bags and transported to the testing site. Plastic cups were weighed and filled with 180 ml. of water each. Sample disks were removed from their protective bags, weighed, placed on top of cups containing water and then secured in place with paraffin wax. A recording of temperature and relative humidity was taken at each 24-hour cycle and noted in appendix I.

Water vapor transmission rate can be calculated as the slope of a graph plotting the weight loss over time and dividing this number by the surface area of the treatment. For the purposes of this study, the mean water vapor transmission rates listed in table 33 are recorded in terms of grams per meters<sup>2</sup> per 24 hours. This experiment indicates that the rate of water vapor transmission is similar for all adhesive formulations tested.



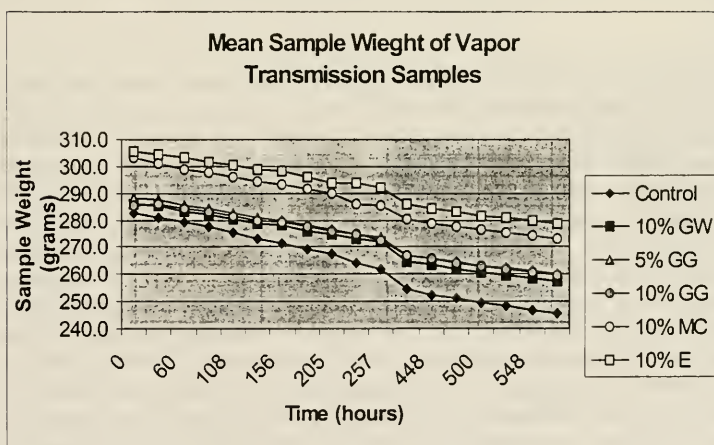
TABLE 33

## Water Vapor Transmission – Summary of Test Data

Sample	Weight Change for Sample 1 (g)	Weight Change for Sample 2 (g)	Weight Change for Sample 3 (g)	Mean Weight Change (g)	Time (h)	Surface Area (m <sup>2</sup> )	Water Vapor Transmission Rate
Control	34.6	41.3	35.1	37.0	572	0.005	12.9
10% GW	27.0	30.3	28.8	28.7	572	0.005	10.0
5% GG	25.6	31.7	26.5	27.9	572	0.005	9.8
10% GG	21.2	29.2	26.0	25.5	572	0.005	8.9
10% MC	33.5	31.9	26.9	30.8	572	0.005	10.8
10% E	24.9	28.0	27.7	26.9	572	0.005	10.2

TABLE 34

## Average Water Vapor Transmission Rates





## Chapter 13

### REVERSABILITY

The principle of reversibility is essential in the selection of any stabilization treatment performed by responsible conservators. This is a difficult criterion to attain since the very purpose of treating an object is to change its natural course of deterioration. As stated by Appelbaum, reversibility

does not require that the object be identical to what it was, only that we can return it to a state where our treatment choices are as broad as they were before the treatment in question was performed.<sup>113</sup>

Future technological innovations are likely to provide new and better methods for both stabilization and analysis. Yet, this must be balanced with current threats to the resource.

Even if an internal consolidant is easily soluble, it is unlikely that much can ever be removed, particularly since objects that need consolidation are by definition so weak that repeated applications of solvent may cause damage...Even a minor treatment like injecting warm gelatin under loose flakes of paint is not reversible, as there is no physical access to the gelatin lodged between the layers of paint after the paint is laid down...An equally important question is: what will happen when the piece needs treatment again, particularly if the problem that necessitated the treatment recurs? Can the same treatment be repeated? Can a different material be used with the first one still in place? What can be done with written condition and treatment records to make it more likely that a future conservator can find out what was done? The undertaking of an admittedly irreversible treatment does not absolve the conservator of responsibility for the future of the object, but increases the importance of a factor we might call, for want of a more elegant term, "re-treatability." The notion of re-treatability

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<sup>113</sup> Barbara Appelbaum. "Criteria for Treatment: Reversability", *Journal of the American Institute for Conservation*, Volume 26, Number 2, Article 1, 1987, p. 65-73.





is one that is often more helpful in evaluating treatments than the idea of reversibility itself. This is particularly true in the impregnation of badly deteriorated materials, since the treatment strengthens what is left of the object but may not prevent further deterioration of original material, and re-treatment may not be far in the future.<sup>114</sup>

Animal proteins have been used for years in the field of objects conservation. Thus, the methods for their retreatment are well documented. Hide glue was used in a variety of applications; possibly the most well documented is in the priming of canvases with Gesso prior to the application of oil paint. The main problem experienced with gelatin adhesive solutions is that they become brittle with age. As the gelatin becomes more brittle, it loses adhesion. Materials testing of adhesives for the consolidation of oil paintings has found that the bond strength is less for retreatment with gelatin solutions to a gelatin based gesso substrate than it is with acrylic solutions to a gelatin based gesso substrate.

Removal of adhesive solutions can be a complex issue. Even in cases where the adhesive formulation is readily soluble, that does not damage either the paint or the substrate, dissolving the adhesive after it has been applied requires heat and agitation. Agitation is impossible once this adhesive has been applied to in-situ. Without agitation, treated areas would require heat and water to melt the gelatin. Unfortunately, heat and water would subject the gelatin to swelling and would reactivating the earthen finishes. Therefore, the removal of gelatin solutions may cause further damage to the earthen surface finishes.

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<sup>114</sup> Barbara Appelbaum, "Criteria for Treatment: Reversability", *Journal of the American Institute for Conservation*, Volume 26, Number 2, Article 1, 1987, p. 65-73.



While conducting these treatments, conservators have developed methods to reduce the potential affects of reverse migration. By rewetting original plasters, carbon may migrate to the surface, appearing as stains on the outermost surface finishes. This affect can be seen in the field when the water from treatment applications result in dark stains on the surface of the plaster. In 2000, two interns working for the University of Pennsylvania developed a method for countering this affect. An application of cyclododocane is currently used to block the migration of such solutions from reaching the surface of the earthen finishes.<sup>115</sup>

However, it should be noted that since gelatin is a biodegradable adhesive that becomes brittle with age, treated areas will eventually lose their adhesion. Other studies have cited that retreatment with gelatin does not provide a bond with the same strength as was originally established. Thus, though retreatment is feasible, further testing needs to be done to establish the best method for retreatment.

Numerous wet chemical methods have been developed to determine the presence of proteinacious binders such as the biochemical biuret test, or testing with givestrichloroacetic acid, tanic acid or ninhydrin. The precipitate with 5 % tannic acid is a particularly sensitive test for very dilute solutions of gelatin. X-ray diffraction and differential scanning calorimetry are other methods commonly used to detect protein conformation changes.<sup>116</sup> Testing of original plasters for protein content yielded negative results and personal communications with representatives from the Hopi asserted that no

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<sup>115</sup> Personal communication with Brynn Bender, objects conservator, 2001 field season.

<sup>116</sup> Messier, Paul. "Work In Progress: An Analysis of the Effect of Water on the Cracking of Albumen Photographs", *Topics In Photographic Preservation*, Volume 4, Compiled by Robin E. Siegel, American Institute for Conservation of Photographic Materials Group, 1991.



proteins were mixed into their traditional earthen plasters. Thus, a positive result for protein could indicate that conservation treatment had occurred at this location. When this test yields a positive result, it provides conservators with an additional method to determine the locations of previous treatments.<sup>117</sup> When combined with the detailed method of documentation developed by the University of Pennsylvania, the treatment methods examined in this study would extend the life of this resource without destroying the potential for further study of the resource.

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<sup>117</sup> Watkins, Stephanie. "Chemical Watermarking of Paper", *Journal of the American Institute for Conservation*, Volume 29, Number 2, Article 2, 1990, p. 117-131.



### CONCLUSIONS

In conclusion, it is the responsibility of a conservator to have a good understanding of the cultural resource that they are treating, the mechanisms of deterioration affecting this resource, detailed methods for recording these conditions, an understanding of treatment methods and a monitoring plan. This study has attempted to provide preliminary information necessary to fulfill this responsibility.

A vast quantity of research has been conducted on the topic of stabilization treatments for earthen surface finishes at Mesa Verde National Park. This research has led to the development of a detailed condition survey and monitoring procedure. Previous testing had been conducted in-situ prior to the outset of this survey. Previous field testing involved the use of both acrylic resins and gelatin solutions. This study has provided materials testing data that should be considered when selecting an adhesive suitable for administering earthen finish conservation treatments.

The experiments in this study were conducted to clarify the mechanisms of deterioration that contribute to a loss of Ancestral Puebloan finishes. The ideal adhesive would provide a bond strength between substrate and surface finish that is slightly weaker than the cohesive strength of these two adherents. It would sufficiently bond the adherents but not seal their pore structure, allowing for good vapor penetration and low reflectance. The adhesive formulation would be non-toxic, easy to apply and low cost. The ideal treatment would be acceptable to Native American Tribes. It would be flexible enough to buffer the environmentally induced differential movement at the junction of





both adherents. The ideal treatment would also be easily removed, resistant to environmental degradation, durable, long lasting, and be suitable to accommodate future retreatments.

The criteria listed above are virtually impossible to satisfy. Yet, each of these criteria has been met by at least one of the adhesive formulations tested for this study. Therefore, the question becomes not which formulation is the ideal solution for the treatment of earthen surface finishes, but which solution is best suited to the specific environmental fluctuations that it will be subjected to.

Based on observations made during accelerated weathering experiments and observations made while testing the bond strength of weathered materials, the nature of this deterioration causes a continued decrease in the adhesive strength of these particles. Thus, the cohesive strength of each layer and the adhesive strength of one layer to the next are perpetually weakened by the natural forces of environmentally induced deterioration. Since the strength of original materials is not constant, the ideal adhesive solution would lose adhesive strength at the same rate as the adherents. Thus, a biodegradable adhesive such as gelatin would be preferred. Still, enough information has not yet been collected to determine the rate of deterioration. Thus, continued monitoring of treated areas is essential for the long-term assessment of these treatments.

In this case, adhesive films should be viscous enough to provide good adhesion of particles of both adherents. However, the penetration of an adhesive should be minimal and the wetting of particles not be so thorough that they induce a significant change in reflective index. The high viscosity and wetting ability of acrylic emulsions allow them to be easily injected with a hypodermic syringe, but also allows them to fill pores and



create a uniformly smooth surface. This can result in noticeable changes of the visual properties of surface finishes. The gelatin solutions, though still indictable, were less viscous and easily converted from liquid to gel state. The reduced wettability and penetration of gelatin based adhesives made them less prone to these detrimental changes in optical quality.

The ability of an adhesive to absorb stresses that induced the initial damage is dependent on many factors. The most prominent physical property affecting this ability is elasticity. A sufficiently flexible adhesive film allows the adherents to move independently from one another without losing contact with the adhesive film. Visual examinations of all four solutions revealed that films of the plasticized gelatin retained the most flexibility. As a measure of flexibility, so long as the external plasticizer remains entrapped within the gelatin structure, the plasticized gelatin solution is far better than that of its acrylic counterparts. Though the acrylic solutions were stronger and easier to apply, they did not offer the same elasticity that the externally plasticized gelatin solution can provide. The logical result of such treatment would be a very stable, strong, and well consolidated area that was treated. However, the same fluctuations of environmentally induced expansion and contraction that induced the initial damage would still be active. Thus, instead of stabilizing the entire wall, acrylic emulsions would theoretically stabilize the areas of initial weakness while the same stresses would induce strain-related damage in other portions of the wall when subjected to further environmental fluctuations.

Since the primary environmental factor that induces loss of adhesion is water, vapor transmission rate is another significant criteria when selecting an adhesive solution.



The results of this test showed all formulations to be fairly vapor permeable relative to the control. However, the ability of a gelatin and glycerin solution to shrink and swell when exposed to humidity present concerns for the use of gelatin based adhesives. In an environment with rapid changes in relative humidity, the swelling of gelatin adhesives could induce added strain to an already weakened area. However, based on preliminary observations at Mesa Verde National Park, the climate is dry enough that this is not a substantial concern so long as the gelatin treatments are applied to areas that are not actively wetted due to drainage, exposure or capillary action. Thus, the use of gelatin adhesive is most useful for the treatment of deterioration that is caused by moisture related exposure that is no longer active, but is still subject to further deterioration from wind and mechanical abrasion. Furthermore, the retention of water within the structure of gelatin adhesives also contributes to the flexibility of the adhesive.

One potentially detrimental effect of water retention is that it makes a favorable environment for biological organisms. However, adding biocides such as sulfur dioxide, beta naphthol or pentachlorophenoline could limit the damage induced by this threat. Since manually removing adhesive treatment formulations is unrealistic, the biodegradable gelatin based adhesives have been favored as a naturally reversible treatment.

Bond strength is the most obvious factor to consider when assessing the feasibility of an adhesive solution. For such tests, the bond strength of acrylic solutions was stronger than that of the gelatin solutions. However, the 10% solution of plasticized gelatin was comparable to 5% solutions of acrylics. Observations made while testing the adhesive bond strength of 10% acrylic solutions indicated that their strength was oftentimes stronger than the earthen finishes.



Of the acrylic emulsions tested, Roplex MC-1835 and Roplex E-330 formulations performed similarly. However, visual observations revealed that facsimile earthen surface finishes treated with MC-1834 withstood accelerated weathering slightly better than those treated with E-330. The greater bond strength of the 10% concentrations of acrylic indicates that a very small amount of acrylic is needed to establish an adhesive bond.

The cohesive strength of an unrestrained, unplasticized film of gelatin proved to be stronger than the earthen plasters it was used to adhere. Hence, gelatin solutions that have not been combined with a plasticizing agent are not recommended. The 5% gelatin based formulations did not provide enough bond strength to ensure prolonged durability of such treatments. However, tests revealed that the 5% MC-1835 provided bond strength comparable to that of the 10% plasticized gelatin formulation. Consequently, of the four proteinacious formulations tested, the 10% plasticized gelatin solution was the more practical option to ensure long-term durability of an adhesive solution.

In selecting an adhesive for reattachment of earthen surface finishes at Mesa Verde National Park, gelatin based formulations were selected in favor of acrylics. This decision weighed considerations of bond strength, water vapor permeability, ease of application, optical quality, low toxicity, reduced cost and accessibility of materials. The testing program described in this study reveals the complexity of issues one must contend with when selecting an adhesive formulation for reattachment of earthen surface finishes. Preliminary research to predict the effectiveness of an adhesive treatment should include both materials and product testing. Research and test results generated by this type of research program enable professional architectural conservators to make educated





decisions regarding the formulation, application and feasibility of individual conservation treatments.



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**APPENDIX A:**  
**List of Soil Samples and Collection Proveniences**



# APPENDIX A: List of Soil Samples and Collection Proveniences

Sample Name	Description of Sample	Origin of Sample	Munsell Color
STH-2 (r)	Spalled reddish plaster finish	Interior of Room 24 (Spruce Tree House 5MV640: Brisben 2001)	7.5RY6/4 Light Brown
SQT-10 (r)	Spalled plaster finish that is predominantly reddish	Interior of Kiva D (Square Tower 5MV650: Barnett & Brisben 2001)	7.5YR6/4 Light Brown
SQT-10 (w)	Spalled finish with white wash overlaying reddish plaster	Interior of Kiva D (Square Tower 5MV650: Barnett & Brisben 2001)	7.5YR8/2 Pinkish White overlaying 7.5YR5/4 Brown
SY-1	Ground caliche fragments	Collected by Kathy Fiero in the vicinity of Balcony House	10YR8/2 White
SY-2	Yellowish soil currently used for Mesa Verde stabilization	Purchased from the Ute Reservation Soil Quarry	10YR7/4 Very Pale Brown
SY-3	Reddish soil currently used for Mesa Verde stabilization	Collected by Kathy Fiero in vicinity of mesa top helipad	5YR5/4 Reddish Brown
MR-1	White Kaolinite	Collected by Rebecca Carr in the vicinity of mile marker 12, Main Park Road	7.5YR8/1 White
MR-4	White sandstone	Collected by Rebecca Carr in the vicinity of mile marker 8, Main Park Road	10YR8/1 White



## **APPENDIX B:**

### **Porosity of Sample Substrates - Total Immersion Test**



## APPENDIX B: Porosity of Sample Substrates - Total Immersion Test

### ***MEVE SANDSTONE***

#### **Pre-Testing Weight (Mo):**

Dry Sample A = 326.5 g

Dry Sample B = 108.9 g

Dry Sample C = 291.1 g

#### **Post-Testing Weight (Md) = 241.8 g:**

Dry Sample A = 326.4 g

Dry Sample B = 108.7 g

Dry Sample C = 290.3 g

Sample Mass (Mn)	ST-A	ST-B	ST-C	Mean Values (M max & WAC)
Mass at 5 min.	329.0 g	113.4 g	295.0 g	245.8 g
Change in Mass	0.77%	4.13%	1.34%	0.42%
Mass at 10 min.	329.3 g	114.1 g	296.4 g	246.6 g
Change in Mass	0.86%	4.78%	1.82%	1.74%
Mass at 15 min.	329.4 g	114.3 g	296.8 g	246.8 g
Change in Mass	0.89%	4.96%	1.96%	2.07%
Mass at 30 min.	330.1 g	114.4 g	297.5 g	247.3 g
Change in Mass	1.10%	5.05%	2.20%	2.27%
Mass at 60 min.	331.5 g	114.5 g	298.7 g	248.2 g
Change in Mass	1.53%	5.14%	2.61%	2.65%
Mass at 790 min.	334.5 g	114.5 g	299.9 g	249.6 g
Change in Mass	2.45%	5.14%	3.02%	3.23%
Mass at 1145 min.	334.7 g	114.5 g	300.2 g	249.8 g
Change in Mass	2.51%	5.14%	3.13%	3.31%
Mass at 1440 min.	334.7 g	114.5 g	300.1 g	249.8 g
Change in Mass	2.51%	5.14%	3.09%	3.31%
Mass at 2880 min.	334.7 g	114.5 g	300.9 g	250.0 g
Change in Mass	2.51%	5.14%	3.37%	3.39%
Mass at 4320 min.	335.7 g	115.0 g	300.9 g	250.5 g
Change in Mass	2.82%	5.60%	3.37%	3.60%
Mass at 5770 min.	336.3 g	115.4 g	301.4 g	251.0 g
Change in Mass	3.00%	5.97%	3.54%	3.80%
Mass at 7200 min.	335.6 g	115.4 g	301.4 g	250.8 g
Change in Mass	2.79%	5.97%	3.54%	3.72%
Mass at 8640 min.	335.9 g	115.5 g	301.4 g	250.9 g
Change in Mass	2.88%	6.06%	3.54%	3.76%
Mass at 10200 min.	335.9 g	115.6 g	301.4 g	251.0 g
Change in Mass	2.88%	6.15%	3.54%	3.80%
Hygrostatic weight	201.8 g	67.6 g	180.1 g	149.8 g





## APPENDIX B: Porosity of Sample Substrates - Total Immersion Test

<b>MODERN EXTRUDED BRICK (Penn)</b>				
<b>Pre-Testing Weight (Mo):</b>		<b>Post-Testing Weight (Md) = 193.7 g:</b>		
Dry Sample A = 188.9 g		Dry Sample A = 188.9 g		
Dry Sample B = 185.8 g		Dry Sample B = 185.8 g		
Dry Sample C = 206.4 g		Dry Sample C = 206.4 g		
Sample Mass (Mn)	ST-A	ST-B	ST-C	Mean Values (M max & WAC)
Mass at 5 min.	200.8 g	197.2 g	218.8 g	205.6 g
Change in Mass	6.30%	6.14%	6.01%	6.14%
Mass at 10 min.	200.8 g	197.4 g	218.8 g	205.7 g
Change in Mass	6.30%	6.24%	6.01%	6.20%
Mass at 15 min.	200.8 g	197.4 g	218.8 g	205.7 g
Change in Mass	6.30%	6.24%	6.01%	6.20%
Mass at 30 min.	200.8 g	197.4 g	219.0 g	205.7 g
Change in Mass	6.30%	6.24%	6.30%	6.20%
Mass at 60 min.	201.3 g	197.8 g	219.2 g	206.1 g
Change in Mass	6.56%	6.46%	6.20%	6.40%
Mass at 940 min.	202.2 g	199.2 g	220.2 g	207.2 g
Change in Mass	7.04%	7.21%	6.69%	6.97%
Mass at 1145 min.	202.8 g	199.3 g	221.1 g	207.7 g
Change in Mass	7.36%	7.27%	7.12%	7.23%
Mass at 1480 min.	202.9 g	199.3 g	221.3 g	207.8 g
Change in Mass	7.41%	7.27%	7.22%	7.28%
Mass at 2880 min.	204.2 g	200.9 g	222.6 g	209.2 g
Change in Mass	8.10%	8.13%	7.85%	8.02%
Mass at 4320 min.	204.9 g	201.1 g	223.4 g	209.8 g
Change in Mass	8.47%	8.23%	8.24%	8.31%
Mass at 5760 min.	205.0 g	201.5 g	223.6 g	210.0 g
Change in Mass	8.52%	8.45%	8.33%	8.42%
Mass at 7260 min.	205.3 g	201.6 g	224.0 g	210.3 g
Change in Mass	8.68%	8.50%	8.53%	8.57%
Mass at 8650 min.	205.4 g	201.7 g	224.0 g	210.4 g
Change in Mass	8.73%	8.56%	8.53%	8.60%
Mass at 10100 min.	205.6 g	202.0 g	224.0 g	210.5 g
Change in Mass	8.84%	8.72%	8.53%	8.69%
Hygrostatic weight	111.2 g	109.6 g	121.5 g	114.1 g



## APPENDIX B: Porosity of Sample Substrates - Total Immersion Test

<b><i>HISTORIC BRICK (Yellow New Orleans)</i></b>				
<b><u>Pre-Testing Weight (Mo):</u></b>		<b><u>Post-Testing Weight (Md) = 156.6 g:</u></b>		
Dry Sample A = 92.9 g		Dry Sample A = 92.6 g		
Dry Sample B = 176.3 g		Dry Sample B = 176.0 g		
Dry Sample C = 201.5 g		Dry Sample C = 201.2 g		
Sample Mass (Mn)	ST-A	ST-B	ST-C	Mean Values (M max & WAC)
Mass at 5 min.	110.0 g	209.4 g	237.2 g	185.5 g
Change in Mass	18.41%	18.77%	17.72%	18.45%
Mass at 10 min.	110.1 g	209.3 g	237.3 g	185.6 g
Change in Mass	18.51%	18.72%	17.77%	18.52%
Mass at 15 min.	110.1 g	209.5 g	237.3 g	185.7 g
Change in Mass	18.51%	18.83%	17.77%	18.53%
Mass at 30 min.	110.2 g	209.5 g	237.3 g	185.7 g
Change in Mass	18.62%	18.83%	17.77%	18.53%
Mass at 60 min.	110.5 g	210.4 g	237.6 g	186.2 g
Change in Mass	18.95%	19.34%	17.92%	18.88%
Mass at 620 min.	111.1 g	211.3 g	239.2 g	187.2 g
Change in Mass	19.59%	19.85%	18.71%	19.54%
Mass at 1160 min.	111.8 g	212.5 g	240.1 g	188.1 g
Change in Mass	20.34%	20.53%	19.16%	20.14%
Mass at 1330 min.	111.9 g	212.4 g	240.1 g	188.1 g
Change in Mass	20.45%	20.48%	19.16%	20.14%
Mass at 1480 min.	112.0 g	212.5 g	240.4 g	188.3 g
Change in Mass	20.56%	20.53%	19.31%	20.25%
Mass at 2880 min.	112.9 g	214.3 g	242.4 g	189.9 g
Change in Mass	21.53%	21.55%	20.30%	21.26%
Mass at 4320 min.	113.1 g	214.7 g	243.1 g	190.3 g
Change in Mass	21.74%	21.78%	20.65%	21.52%
Mass at 5760 min.	113.2 g	215.1 g	243.8 g	190.7 g
Change in Mass	21.85%	22.01%	20.99%	21.78%
Mass at 7200 min.	113.2 g	215.2 g	243.8 g	190.7
Change in Mass	21.85%	22.06%	20.99%	21.78%
Hygrostatic weight	50.1 g	94.3 g	106.2 g	83.5 g



## APPENDIX B: Porosity of Sample Substrates - Total Immersion Test

<b><i>HISTORIC BRICK (Red New Orleans)</i></b>				
<b><u>Pre-Testing Weight (Mo):</u></b>		<b><u>Post-Testing Weight (Md) = 171.2 g:</u></b>		
Dry Sample A = 191.7 g		Dry Sample A = 191.4 g		
Dry Sample B = 139.5 g		Dry Sample B = 139.0 g		
Dry Sample C = 183.4 g		Dry Sample C = 183.1 g		
Sample Mass (Mn)	ST-A	ST-B	ST-C	Mean Values (M max & WAC)
Mass at 5 min.	227.3 g	165.1 g	218.0 g	203.5 g
Change in Mass	18.57%	18.35%	18.87%	18.85%
Mass at 10 min.	227.3 g	165.3 g	218.0 g	203.5 g
Change in Mass	18.57%	18.49%	18.87%	18.89%
Mass at 15 min.	227.3 g	165.4 g	218.1 g	203.6 g
Change in Mass	18.57%	18.57%	18.92%	18.93%
Mass at 30 min.	227.3 g	165.4 g	218.1 g	203.6 g
Change in Mass	18.57%	18.56%	18.92%	18.93%
Mass at 60 min.	227.3 g	165.4 g	218.1 g	203.6 g
Change in Mass	18.57%	18.57%	18.92%	18.93%
Mass at 620 min.	228.6 g	166.4 g	219.3 g	204.8 g
Change in Mass	19.25%	19.28%	19.57%	19.63%
Mass at 1160 min.	228.9 g	166.5 g	220.0 g	205.1 g
Change in Mass	19.41%	19.35%	19.96%	19.82%
Mass at 1330 min.	229.2 g	166.6 g	220.2 g	205.3 g
Change in Mass	19.56%	19.35%	20.65%	19.94%
Mass at 1480 min.	229.6 g	166.9 g	220.4 g	205.6 g
Change in Mass	19.77%	19.43%	20.17%	20.11%
Mass at 2880 min.	231.3 g	168.4 g	221.9 g	207.3 g
Change in Mass	20.66%	20.72%	20.99%	21.03%
Mass at 4320 min.	232.4 g	169.2 g	222.7 g	208.1 g
Change in Mass	21.23%	21.29%	21.43%	21.55%
Mass at 5760 min.	234.2 g	170.4 g	223.5 g	209.4 g
Change in Mass	22.17%	22.15%	21.86%	22.29%
Mass at 7200 min.	234.2 g	170.5 g	223.5 g	209.4 g
Change in Mass	22.17%	22.22%	21.86%	22.31%
Hygrostatic weight	111.9 g	81.5 g	106.1 g	99.8 g



## APPENDIX B: Porosity of Sample Substrates - Total Immersion Test

### **MODERN BRICK**

#### **Pre-Testing Weight (Mo):**

Dry Sample A = 225.9 g

Dry Sample B = 236.7 g

#### **Post-Testing Weight (Md) = 231.1 g:**

Dry Sample A = 225.7 g

Dry Sample B = 236.4 g

Sample Mass (Mn)	ST-A	ST-B	Mean Values (M max & WAC)
Mass at 5 min.	238.2 g	249.4 g	243.8 g
Change in Mass	5.44%	5.37%	5.52%
Mass at 10 min.	238.2 g	249.4 g	243.8 g
Change in Mass	5.44%	5.37%	5.52%
Mass at 15 min.	238.3 g	249.4 g	243.9 g
Change in Mass	5.48%	5.37%	5.54%
Mass at 30 min.	238.9 g	249.9 g	244.4 g
Change in Mass	5.75%	5.56%	5.78%
Mass at 60 min.	239.6 g	250.8 g	245.2 g
Change in Mass	6.06%	5.96%	6.12%
Mass at 620 min.	240.1 g	251.9 g	246.0 g
Change in Mass	6.29%	6.42%	6.47%
Mass at 1160 min.	241.4 g	252.3 g	246.9 g
Change in Mass	6.86%	6.59%	6.84%
Mass at 1330 min.	241.4 g	252.5 g	247.0 g
Change in Mass	6.86%	6.68%	6.88%
Mass at 1480 min.	241.6 g	252.8 g	247.2 g
Change in Mass	6.95%	6.80%	6.97%
Mass at 2880 min.	242.3 g	253.5 g	247.9 g
Change in Mass	7.26%	7.10%	7.27%
Mass at 4320 min.	242.6 g	253.5 g	248.1 g
Change in Mass	7.39%	7.10%	7.33%
Mass at 5760 min.	242.8 g	254.0 g	248.4 g
Change in Mass	7.48%	7.31%	7.49%
Mass at 7200 min.	242.8 g	253.9 g	248.4 g
Change in Mass	7.48%	7.27%	7.49%
Hygrostatic weight	138.2 g	144.6 g	141.4 g



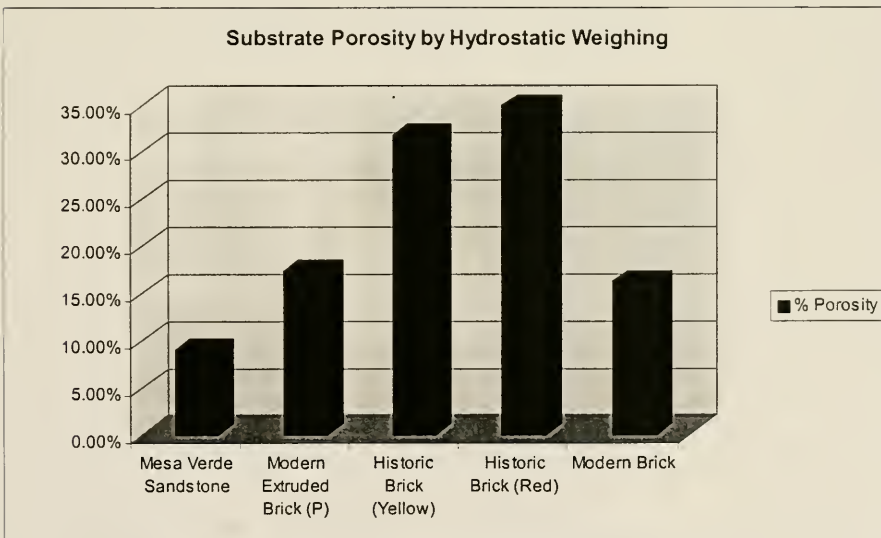


**APPENDIX C:**  
**Porosity of Sample Substrates - Hygrostatic Weighing Test**



### APPENDIX C: Porosity of Sample Substrates - Hydrostatic Weighing Test

	Sandstone	Modern Extruded Brick (P)	Historic Brick (Yellow)	Historic Brick (Red)	Modern Brick
Dry Mass (M1)	241.8	193.7	156.6	171.2	231.1
Immersed Mass (M2)	149.8	114.1	83.5	99.8	141.4
Saturated Mass (M3)	251.0	210.5	190.7	209.4	248.4
Pore Volume ( $V_p = M3 - M1$ )	9.2	16.8	34.1	38.2	17.3
Apparent Volume ( $V_a = M3 - M2$ )	101.2	96.4	107.2	109.6	107
Real Volume ( $V_r = M1 - M2$ )	92	79.6	73.1	71.4	89.7
Real Density ( $Pr = [M1 / V_r] \times 10^3$ )	2628.3	2433.4	2142.3	2397.8	2576.4
Apparent Density ( $Pa = [M1 / V_a] \times 10^3$ )	2389.3	2009.3	1460.8	1562.0	2159.8
% Porosity ( $[(1 - Pa / Pr) \times 100]$ )	9.0%	17.4%	31.8%	34.9%	16.2%





**APPENDIX D:**  
**Common Additives used in Gelatin Based Formulations**



## APPENDIX D: Common Additives used in Gelatin Based Formulations

Material Specifications		Description	Use
Phenol (monohydroxy benzene)		A byproduct in the processing of acetone. Phenols have a hydroxyl (-OH) group bonded to a carbon atom that forms part of an aromatic ring. Phenols form stronger hydrogen bonds than alcohols, are water soluble, and acidic, and react with formaldehyde. P-phenyl phenol is commonly used in gelatin solutions.	Preservative <sup>118</sup>
Chemical Formula	C <sub>6</sub> H <sub>6</sub> O		(not recommended for use on alkaline surfaces)
Molecular Weight:	94.1128		
Formaldehyde		A gas that is usually sold diluted in 37%–40% water and sold as “formalin”. Often combined with gelatin and gum arabic. Hardens the surface of gelatin paints when sprayed on the surface after cure. <sup>119</sup> Also used to make gelatin capsules for pharmaceutical drugs.	Hardener, preservative, decreases water solubility
Chemical Formula	CH <sub>2</sub> O		
Molecular Weight:	30.03		
Glutaraldehyde		Cross-links with gelatin and gum arabic in solution. Commonly used to make gelatin capsules for pharmaceutical drugs.	Hardener, improves thermal resistance, decreases water solubility
Chemical Formula	C <sub>5</sub> H <sub>8</sub> O <sub>2</sub>		
Molecular Weight:	100.12		
Alum (Potassium chrome alum) <sup>120</sup>		A potassium aluminum sulfate salt. Used for paper making and leather tanning. <sup>121</sup>	Hardener, decreases water solubility
Chemical Formula	KAl(SO <sub>4</sub> ) <sub>2</sub> ·12H <sub>2</sub> O		
Molecular Weight:	788.13 <sup>122</sup>		

<sup>118</sup> Cassar, JoAnn and Roberta de Angelis. “Glossary. Materials used in 19th and 20th century Plaster Architecture. Plaster Architecture Essay, Culture 2000 Programme of the European Union.

<http://www.plasterarc.net/essay/essay/CassarG.html>

<sup>119</sup> Jusko, Don A. “History of Painting Mediums... Glue, Wax Paint, Cera Colla, Mastic, Casein Paint, Fresco, Egg, Oil Paint, Acrylic Paint”, mauigateway.com.

<http://www.mauigateway.com/~donjusko/1mediums.htm#100,000 B/C>

<sup>120</sup> Cassar, JoAnn and Roberta de Angelis. “Glossary. Materials used in 19th and 20th century Plaster Architecture. Plaster Architecture Essay, Culture 2000 Programme of the European Union.

<http://www.plasterarc.net/essay/essay/CassarG.html>

<sup>121</sup> Merriam-Webster Inc. “Dictionary”, <http://www.webster.com>

<sup>122</sup> Krompiec, Michal and Luc Patiny. “ChemCalc”, Silesian University of Technology, University of Lausanne, 5<sup>th</sup> International Electronic Conference on synthetic Organic Chemistry, ChemExper.

<http://www.chemcalc.org/>





## APPENDIX D: Common Additives used in Gelatin Based Formulations

Material Specifications		Description	Use
Sorbitol		A hexahydric alcohol that is used as a sweetener in food products <sup>123</sup> , as a plasticizer in glue formulations <sup>124</sup> and as a softening agent for textiles, paper and leather. <sup>125</sup> It occurs naturally, but is commercially produced by reducing aqueous glucose solutions as well.	Plasticizer  (Not recommended for use in arid climates)
Chemical Formula	C <sub>6</sub> H <sub>14</sub> O <sub>6</sub>		
Molecular Weight:	182.17		
Ammonia		A colorless, alkaline gas that can be condensed into liquid form with cold temperature and pressure. <sup>126</sup>	Modifies the pH of gelatin formulations. <sup>127</sup>
Chemical Formula	NH <sub>3</sub>		
Molecular Weight:	17.03		
Sulphur Dioxide		Sulfur dioxide is a gas that is used as a solvent in paper production. Sulfur is also used as a fungicide and insecticide. It is formed by burning sulfur.	Preservative, bleaching agent.
Chemical Formula	SO <sub>2</sub>		
Molecular Weight:	64.06		
Diethylene Glycol (DEG)		Hygroscopic liquid that is used in the production of polyester resins and polyurethanes. It is also used in the production of paper, glue and cellophane.	Plasticizer
Chemical Formula	C <sub>4</sub> H <sub>10</sub> O <sub>3</sub>		
Molecular Weight:	106.12		
Iso-Propyl Alcohol (Isopropanol)		Used as a wetting agent and a solvent in many paint formulations and as a surfactant in cleaning solutions. It also has in medicinal and herbicidal (glyphosate) uses. Isopropyl is similar to ethyl alcohol in solvent properties.	Wetting agent
Chemical Formula	C <sub>3</sub> H <sub>8</sub> O		
Molecular Weight:	60.09		

<sup>123</sup> Partos, Lindsey. "FoodNavigator", 2000/2002. <http://www.foodnavigator.com/internet/bestof.asp>

<sup>124</sup> Cassar, JoAnn and Roberta de Angelis. "Glossary. Materials used in 19th and 20th century Plaster Architecture. Plaster Architecture Essay, Culture 2000 Programme of the European Union. <http://www.plasterarc.net/essay/essay/CassarG.html>

<sup>125</sup> Matt T. Roberts, Don Etherington and Margaret R. Brown. "Bookbinding and the Conservation of books: A Dictionary of Descriptive Terminology", Stanford University Libraries, 1994. <http://palimpsest.stanford.edu/don/don.html>

<sup>126</sup> Merriam-Webster Inc. "Dictionary", <http://www.webster.com>

<sup>127</sup> Cole, Bernard. "Gelatin Food Science", University of Pretoria, <http://www.gelatin.co.za/gltm1.html>



## APPENDIX D: Common Additives used in Gelatin Based Formulations

Material Specifications		Description	Use
Glycerol (Glycerin)		A byproduct in the manufacture of animal fats and plant oils into soap. Glycerin is a hygroscopic trihedric alcohol. Due to its hygroscopicity, it is used to retain moisture in cosmetics and chemically reacts with acetic anhydrides.	Plasticizer, wetting agent, antifreeze
Chemical Formula	$C_3H_5(OH)_3$		
Molecular Weight:	92.09		
Propylene glycol		Used as an antifreeze and solvent. Cellulose based glycols are also used as plasticizers in glues. <sup>128</sup>	Solvent, wetting agent, plasticizer
Chemical Formula	$C_3H_8O_2$		
Molecular Weight:	76.10		
Gelatin (Collagen Protein)		Protein made from denatured collagen that has been hydrolized by either heat or changes in pH. There is variation in the composition and pH depending on manufacturing processes. Thus, an approximate chemical formula and molecular weight are given. <sup>129</sup>	Adhesive, defoaming agent, gelling agent
Chemical Formula	$C_{102}H_{151}O_{39}N_{31}$		
Molecular Weight:	2435.51		
Beta naphthol		A monohydric alcohol within the phenol family. It is manufactured by fusing naphthalenesulfonic acid with caustic soda and is used as biocidic preservative in gelatin solutions. It is also used in leather tanning, antioxidants, and antiseptics. <sup>130</sup>	Preservative
Chemical Formula	$C_{10}H_7OH$		
Molecular Weight:	144.17		
Pentachlorophenoline		A crystalline compound that found in rabbit skin glue solutions as a preservative. Also used as a wood preservative.	Preservative
Chemical Formula	$C_6Cl_5OH$		
Molecular Weight:	266.34		

<sup>128</sup> Matt T. Roberts and Don Etherington. Bookbinding and the Conservation of books: A Dictionary of Descriptive Terminology <http://palimpsest.stanford.edu/don/don.html>

<sup>129</sup> Matt T. Roberts and Don Etherington. Bookbinding and the Conservation of books: A Dictionary of Descriptive Terminology <http://palimpsest.stanford.edu/don/don.html>

<sup>130</sup> Chemical Land 21.com Arokor Holdings Inc. 2000. [www.chemicalland21.com](http://www.chemicalland21.com)



## **APPENDIX E:**

### **Viscosity of Acrylic and Gelatin Adhesive Solutions**



# APPENDIX E: Viscosity of Acrylic and Gelatin Adhesive Solutions

Concentration of Solution	Cup Size	Temperature (Celsius)	Time (Seconds)	Time (Seconds)	Time (Seconds)	MEAN VISCOSITY
5% GW	#1	25	35.3	36.1	32.8	34.7
5% GW	#1	48	31.5	31.6	31.4	31.5
5% GW	#2	25	16.1	15.6	15.8	15.8
5% GW	#3	25	7.6	8.2	8.1	8.0
10% GW	#1	25	50.8	56.1	52.3	53.1
10% GW	#1	51	35.2	35.4	35.2	35.3
10% GW	#1	61	33.9	34.3	34.1	34.1
10% GW	#2	25	28.0	23.9	26.7	26.2
10%GW	#3	29	8.3	8.4	8.3	8.3
5% GG	#1	25	35.8	40.4	38.7	38.3
5%GG	#1	56	30.5	30.7	31.0	30.7
5%GG	#1	70	30.8	31.2	31.2	31.1
5% GG	#2	25	24.8	27.4	27.6	26.6
10% GG	#1	27	45.5	44.8	46.2	45.5
10%GG	#1	47	33.3	37.4	37.4	36.0
10%GG	#1	66	34.5	34.4	34.6	34.5
10% GG	#2	25	23.9	24.6	25.3	24.6
10% GG	#3	25	16.3	15.7	17.2	16.4
5% MC	#1	25	28.5	28.6	28.8	28.6
5% MC	#2	25	13.8	14.1	13.9	13.9
10% MC	#1	25	28.6	28.5	28.5	28.5
10% MC	#2	25	13.8	12.9	13.6	13.4
5% E	#1	25	28.7	28.8	29.0	28.8
5% E	#2	25	13.7	13.9	13.8	13.8
10% E	#1	25	29.4	28.9	29.1	29.1
10% E	#2	25	13.8	13.9	13.7	13.8





## **APPENDIX F:**

### **Shrinkage Cracks formed on Reproduction Surface Finishes**



## APPENDIX F: Shrinkage Cracks formed on Reproduction Surface Finishes

Sample #	Substrate	Treatment Solution	Temperature (Celsius)	Observations (24 hours)
RBP-1	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	25	Micro-Cracks
RBP-2	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	25	Micro-Cracks
RBP-3	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	25	Micro-Cracks
RBP-4	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	25	No Cracks; still in gel state on top of surface
RBP-5	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	25	No Cracks; still in gel state on top of surface
RBP-6	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	25	No Cracks; still in gel state on top of surface
RBP-7	Reddish Brown Plaster (60:40)	5% Gelatin in Water	25	Micro-Cracks
RBP-8	Reddish Brown Plaster (60:40)	5% Gelatin in Water	25	No Cracks
RBP-9	Reddish Brown Plaster (60:40)	5% Gelatin in Water	25	Micro-cracks
RBP-10	Reddish Brown Plaster (60:40)	10% Gelatin in Water	25	Detached
RBP-11	Reddish Brown Plaster (60:40)	10% Gelatin in Water	25	Detached
RBP-12	Reddish Brown Plaster (60:40)	10% Gelatin in Water	25	Detached
RBP-13	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	30	Micro-Cracks



# APPENDIX F: Shrinkage Cracks formed on Reproduction Surface Finishes

Sample #	Substrate	Treatment Solution	Temperature (Celsius)	Observations (24 hours)
RBP-14	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	30	Micro-Cracks
RBP-15	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	30	Micro-Cracks
RBP-16	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	30	No Cracks
RBP-17	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	30	No Cracks
RBP-18	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	30	Macro-Cracks
RBP-19	Reddish Brown Plaster (60:40)	5% Gelatin in Water	30	Micro-Cracks
RBP-20	Reddish Brown Plaster (60:40)	5% Gelatin in Water	30	Micro-Cracks
RBP-21	Reddish Brown Plaster (60:40)	5% Gelatin in Water	30	Micro-Cracks
RBP-22	Reddish Brown Plaster (60:40)	10% Gelatin in Water	30	Micro-Cracks
RBP-23	Reddish Brown Plaster (60:40)	10% Gelatin in Water	30	Macro-Cracks
RBP-24	Reddish Brown Plaster (60:40)	10% Gelatin in Water	30	Macro-Cracks
WW-1	White (SY-1) Wash (10:90)	5% Gelatin with Glycerin in Water	25	Micro-Cracks
WW-2	White (SY-1) Wash (10:90)	5% Gelatin with Glycerin in Water	25	Micro-Cracks
WW-3	White (SY-1) Wash (10:90)	5% Gelatin with Glycerin in Water	25	Micro-Cracks
WW-4	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	25	No Cracks; still in gel state on top of surface



## APPENDIX F: Shrinkage Cracks formed on Reproduction Surface Finishes

Sample #	Substrate	Treatment Solution	Temperature (Celsius)	Observations (24 hours)
WW-5	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	25	No Cracks; still in gel state on top of surface
WW-6	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	25	No Cracks; still in gel state on top of surface
WW-7	White (SY-1) Wash (10:90)	5% Gelatin in Water	25	Micro-Cracks
WW-8	White (SY-1) Wash (10:90)	5% Gelatin in Water	25	Micro-Cracks
WW-9	White (SY-1) Wash (10:90)	5% Gelatin in Water	25	No Cracks
WW-10	White (SY-1) Wash (10:90)	10% Gelatin in Water	25	Detached
WW-11	White (SY-1) Wash (10:90)	10% Gelatin in Water	25	Detached
WW-12	White (SY-1) Wash (10:90)	10% Gelatin in Water	25	Detached
WW-13	White (SY-1) Wash (10:90)	5% Gelatin with Glycerin in Water	30	Micro-Cracks
WW-14	White (SY-1) Wash (10:90)	5% Gelatin with Glycerin in Water	30	Micro-Cracks
WW-15	White (SY-1) Wash (10:90)	5% Gelatin with Glycerin in Water	30	Micro-Cracks
WW-16	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	30	No Cracks
WW-17	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	30	No Cracks
WW-18	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	30	No Cracks
WW-19	White (SY-1) Wash (10:90)	5% Gelatin in Water	30	No Cracks
WW-20	White (SY-1) Wash (10:90)	5% Gelatin in Water	30	Micro-Cracks
WW-21	White (SY-1) Wash (10:90)	5% Gelatin in Water	30	Micro-Cracks
WW-22	White (SY-1) Wash (10:90)	10% Gelatin in Water	30	Detached to underlying layer
WW-23	White (SY-1) Wash (10:90)	10% Gelatin in Water	30	Detached





## APPENDIX F: Shrinkage Cracks formed on Reproduction Surface Finishes

Sample #	Substrate	Treatment Solution	Temperature (Celsius)	Observations (24 hours)
WW-24	White (SY-1) Wash (10:90)	10% Gelatin in Water	30	Detached
RBP-25	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	40	No Cracks
RBP-26	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	40	Micro-Cracks
RBP-27	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	40	Micro-Cracks
RBP-28	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	40	Micro-Cracks
RBP-29	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	40	Micro-Cracks
RBP-30	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	40	Micro-Cracks
RBP-31	Reddish Brown Plaster (60:40)	5% Gelatin in Water	40	Micro-Cracks
RBP-32	Reddish Brown Plaster (60:40)	5% Gelatin in Water	40	No Cracks
RBP-33	Reddish Brown Plaster (60:40)	5% Gelatin in Water	40	Micro-Cracks
RBP-34	Reddish Brown Plaster (60:40)	10% Gelatin in Water	40	Micro-Cracks
RBP-35	Reddish Brown Plaster (60:40)	10% Gelatin in Water	40	Micro-Cracks
RBP-36	Reddish Brown Plaster (60:40)	10% Gelatin in Water	40	Micro-Cracks
RBP-37	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	20	No Cracks



## APPENDIX F: Shrinkage Cracks formed on Reproduction Surface Finishes

Sample #	Substrate	Treatment Solution	Temperature (Celsius)	Observations (24 hours)
RBP-38	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	20	No Cracks
RBP-39	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	20	No Cracks
RBP-40	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	20	No Cracks
RBP-41	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	20	No Cracks
RBP-42	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	20	No cracks
RBP-43	Reddish Brown Plaster (60:40)	5% Gelatin in Water	20	Detached
RBP-44	Reddish Brown Plaster (60:40)	5% Gelatin in Water	20	Detached
RBP-45	Reddish Brown Plaster (60:40)	5% Gelatin in Water	20	Detached
RBP-46	Reddish Brown Plaster (60:40)	10% Gelatin in Water	20	Detached
RBP-47	Reddish Brown Plaster (60:40)	10% Gelatin in Water	20	Detached
RBP-48	Reddish Brown Plaster (60:40)	10% Gelatin in Water	20	Detached
WW-25	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	50	No Cracks
WW-26	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	20	Micro-Cracks
WW-27	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	19	Macro-Cracks
WW-28	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	19	Macro-Cracks
WW-29	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	19	Macro-Cracks



## **APPENDIX G:**

### **Relative Absorption for Reproduction Surface Finishes**



# APPENDIX G: Relative Absorption for Reproduction Surface Finishes

Sample	Absorption Time (UW)	Absorption Time (W)
Reddish Brown Plaster - 1	0.5	2
Reddish Brown Plaster - 2	0.4	2
Reddish Brown Plaster - 3	0.5	2
White Sandstone Wash - 1	0.7	3
White Sandstone Wash - 2	0.5	3
White Sandstone Wash - 3	0.4	3
Reddish Brown Wash - 1	0.5	1
Reddish Brown Wash - 2	0.3	1
Reddish Brown wash - 3	0.4	1
Reddish Brown Plaster covered by White Wash - 1	1.4	2
Reddish Brown Plaster covered by White Wash - 2	0.9	2
Reddish Brown Plaster covered by White Wash - 3	1.0	2
Reddish Brown Plaster covered by Reddish Brown Wash - 1	0.4	2
Reddish Brown Plaster covered by Reddish Brown Wash - 2	0.2	2
Reddish Brown Plaster covered by Reddish Brown Wash - 3	0.2	2
Reddish Brown Plaster and Caliche White Wash Treated with 10%GW - 1	1.5	1.3
Reddish Brown Plaster and Caliche White Wash Treated with 10%GW - 2	1.6	1.4
Reddish Brown Plaster and Caliche White Wash Treated with 10%GW - 3	1.3	1.7
Reddish Brown Plaster and Caliche White Wash Treated with Water - 1	1.4	1.5
Reddish Brown Plaster and Caliche White Wash Treated with Water - 2	0.9	1.3
Reddish Brown Plaster and Caliche White Wash Treated with Water - 3	1.2	1.4
Reddish Brown Plaster and Caliche White Wash Treated with 5%GW - 1	1.1	1.0
Reddish Brown Plaster and Caliche White Wash Treated with 5%GW - 2	0.7	1.0
Reddish Brown Plaster and Caliche White Wash Treated with 5%GW - 3	0.7	1.1
Reddish Brown Plaster and Caliche White Wash Treated with 5%GG - 1	1.4	1.5
Reddish Brown Plaster and Caliche White Wash Treated with 5%GG - 2	0.8	1.4
Reddish Brown Plaster and Caliche White Wash Treated with 5%GG - 3	1.3	1.5
Reddish Brown Plaster and Caliche White Wash Treated with 10%GG - 1	1.0	1.9
Reddish Brown Plaster and Caliche White Wash Treated with 10%GG - 2	1.6	1.7
Reddish Brown Plaster and Caliche White Wash Treated with 10%GG - 3	1.4	5.6





**APPENDIX H:**  
**Cohesive Shrinkage for Gelatin Based Adhesive Formulations**



## APPENDIX H: Cohesive Shrinkage for Gelatin Based Adhesive Formulations

Sample #	Substrate	Treatment Solution	Temperature (Celsius)	Observations (24 hours)
RBP-1	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	25	Micro-Cracks
RBP-2	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	25	Micro-Cracks
RBP-3	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	25	Micro-Cracks
RBP-4	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	25	No Cracks; still in gel state on top of surface
RBP-5	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	25	No Cracks; still in gel state on top of surface
RBP-6	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	25	No Cracks; still in gel state on top of surface
RBP-7	Reddish Brown Plaster (60:40)	5% Gelatin in Water	25	Micro-Cracks
RBP-8	Reddish Brown Plaster (60:40)	5% Gelatin in Water	25	No Cracks
RBP-9	Reddish Brown Plaster (60:40)	5% Gelatin in Water	25	Micro-cracks
RBP-10	Reddish Brown Plaster (60:40)	10% Gelatin in Water	25	Detached
RBP-11	Reddish Brown Plaster (60:40)	10% Gelatin in Water	25	Detached
RBP-12	Reddish Brown Plaster (60:40)	10% Gelatin in Water	25	Detached
RBP-13	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	30	Micro-Cracks



## APPENDIX H: Cohesive Shrinkage for Gelatin Based Adhesive Formulations

Sample #	Substrate	Treatment Solution	Temperature (Celsius)	Observations (24 hours)
RBP-14	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	30	Micro-Cracks
RBP-15	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	30	Micro-Cracks
RBP-16	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	30	No Cracks
RBP-17	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	30	No Cracks
RBP-18	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	30	Macro-Cracks
RBP-19	Reddish Brown Plaster (60:40)	5% Gelatin in Water	30	Micro-Cracks
RBP-20	Reddish Brown Plaster (60:40)	5% Gelatin in Water	30	Micro-Cracks
RBP-21	Reddish Brown Plaster (60:40)	5% Gelatin in Water	30	Micro-Cracks
RBP-22	Reddish Brown Plaster (60:40)	10% Gelatin in Water	30	Micro-Cracks
RBP-23	Reddish Brown Plaster (60:40)	10% Gelatin in Water	30	Macro-Cracks
RBP-24	Reddish Brown Plaster (60:40)	10% Gelatin in Water	30	Macro-Cracks
WW-1	White (SY-1) Wash (10:90)	5% Gelatin with Glycerin in Water	25	Micro-Cracks
WW-2	White (SY-1) Wash (10:90)	5% Gelatin with Glycerin in Water	25	Micro-Cracks
WW-3	White (SY-1) Wash (10:90)	5% Gelatin with Glycerin in Water	25	Micro-Cracks



## APPENDIX H: Cohesive Shrinkage for Gelatin Based Adhesive Formulations

Sample #	Substrate	Treatment Solution	Temperature (Celsius)	Observations (24 hours)
WW-4	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	25	No Cracks; still in gel state on top of surface
WW-5	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	25	
WW-6	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	25	
WW-7	White (SY-1) Wash (10:90)	5% Gelatin in Water	25	Micro-Cracks
WW-8	White (SY-1) Wash (10:90)	5% Gelatin in Water	25	Micro-Cracks
WW-9	White (SY-1) Wash (10:90)	5% Gelatin in Water	25	No Cracks
WW-10	White (SY-1) Wash (10:90)	10% Gelatin in Water	25	Detached
WW-11	White (SY-1) Wash (10:90)	10% Gelatin in Water	25	Detached
WW-12	White (SY-1) Wash (10:90)	10% Gelatin in Water	25	Detached
WW-13	White (SY-1) Wash (10:90)	5% Gelatin with Glycerin in Water	30	Micro-Cracks
WW-14	White (SY-1) Wash (10:90)	5% Gelatin with Glycerin in Water	30	Micro-Cracks
WW-15	White (SY-1) Wash (10:90)	5% Gelatin with Glycerin in Water	30	Micro-Cracks
WW-16	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	30	No Cracks
WW-17	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	30	No Cracks
WW-18	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	30	No Cracks
WW-19	White (SY-1) Wash (10:90)	5% Gelatin in Water	30	No Cracks
WW-20	White (SY-1) Wash (10:90)	5% Gelatin in Water	30	Micro-Cracks
WW-21	White (SY-1) Wash (10:90)	5% Gelatin in Water	30	Micro-Cracks





## APPENDIX H: Cohesive Shrinkage for Gelatin Based Adhesive Formulations

Sample #	Substrate	Treatment Solution	Temperature (Celsius)	Observations (24 hours)
WW-22	White (SY-1) Wash (10:90)	10% Gelatin in Water	30	Detached to underlying layer
WW-23	White (SY-1) Wash (10:90)	10% Gelatin in Water	30	Detached
WW-24	White (SY-1) Wash (10:90)	10% Gelatin in Water	30	Detached
RBP-25	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	40	No Cracks
RBP-26	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	40	Micro-Cracks
RBP-27	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	40	Micro-Cracks
RBP-28	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	40	Micro-Cracks
RBP-29	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	40	Micro-Cracks
RBP-30	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	40	Micro-Cracks
RBP-31	Reddish Brown Plaster (60:40)	5% Gelatin in Water	40	Micro-Cracks
RBP-32	Reddish Brown Plaster (60:40)	5% Gelatin in Water	40	No Cracks
RBP-33	Reddish Brown Plaster (60:40)	5% Gelatin in Water	40	Micro-Cracks
RBP-34	Reddish Brown Plaster (60:40)	10% Gelatin in Water	40	Micro-Cracks
RBP-35	Reddish Brown Plaster (60:40)	10% Gelatin in Water	40	Micro-Cracks



## APPENDIX H: Cohesive Shrinkage for Gelatin Based Adhesive Formulations

Sample #	Substrate	Treatment Solution	Temperature (Celsius)	Observations (24 hours)
RBP-36	Reddish Brown Plaster (60:40)	10% Gelatin in Water	40	Micro-Cracks
RBP-37	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	20	No Cracks
RBP-38	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	20	No Cracks
RBP-39	Reddish Brown Plaster (60:40)	5% Gelatin with Glycerin in Water	20	No Cracks
RBP-40	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	20	No Cracks
RBP-41	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	20	No Cracks
RBP-42	Reddish Brown Plaster (60:40)	10% Gelatin with Glycerin in Water	20	No cracks
RBP-43	Reddish Brown Plaster (60:40)	5% Gelatin in Water	20	Detached
RBP-44	Reddish Brown Plaster (60:40)	5% Gelatin in Water	20	Detached
RBP-45	Reddish Brown Plaster (60:40)	5% Gelatin in Water	20	Detached
RBP-46	Reddish Brown Plaster (60:40)	10% Gelatin in Water	20	Detached
RBP-47	Reddish Brown Plaster (60:40)	10% Gelatin in Water	20	Detached
RBP-48	Reddish Brown Plaster (60:40)	10% Gelatin in Water	20	Detached
WW-25	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	50	No Cracks
WW-26	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	20	Micro-Cracks



#### APPENDIX H: Cohesive Shrinkage for Gelatin Based Adhesive Formulations

Sample #	Substrate	Treatment Solution	Temperature (Celsius)	Observations (24 hours)
WW-27	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	19	Macro-Cracks
WW-28	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	19	Macro-Cracks
WW-29	White (SY-1) Wash (10:90)	10% Gelatin with Glycerin in Water	19	Macro-Cracks



**APPENDIX I:**  
**Adhesive Bond Strength of Treated Samples**





# APPENDIX I: Adhesive Bond Strength of Treated Samples

Sample	Adhesive	Notes	Bond Strength (lbs.)
A	Water	Force Meter Test – break located at interface between substrate and plaster	1.46
J	Water	Force Meter Test – break located at interface between substrate and plaster	1.90
S	Water	Force Meter Test – break located at interface between substrate and plaster	2.35
W	5% Gelatin in Water	Force Meter Test – break located at interface between substrate and plaster	0.10
N	5% Gelatin in Water	Force Meter Test – break located at interface between substrate and plaster	0.04
E	5% Gelatin in Water	Instron test – break located at the interface between substrate and plaster. Scale could not capture exact moment of break.	NA
F	10% Gelatin in Water	Instron test – break located within plaster layer, above the interface between substrate and plaster. Crumbled fracture.	430
P	10% Gelatin in Water	Instron test – break located within plaster layer, above the interface between substrate and plaster. Crumbled fracture.	420
X	10% Gelatin in Water	Instron test – break located within plaster layer, above the interface between substrate and plaster. Crumbled fracture.	442
M	5% Gelatin & 10% Glycerin in Water	Force Meter Test – break located at interface between substrate and plaster	0.07
D	5% Gelatin & 10% Glycerin in Water	Force Meter Test – break located at interface between substrate and plaster	0.43
V	5% Gelatin & 10% Glycerin in Water	Force Meter Test – break located at interface between substrate and plaster	0.12
G	10% Gelatin & 10% Glycerin in Water	Instron test – break located at the interface between substrate and plaster Lost failure point. Crumbled fracture.	286
Z	10% Gelatin & 10% Glycerin in Water	Instron test – half of the break was located at the interface between substrate and plaster while the other half crumbled but did not fully detach from the substrate.	125



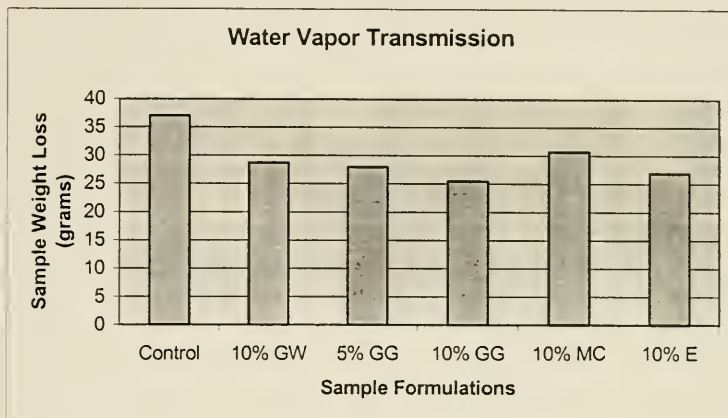
**APPENDIX I: Adhesive Bond Strength of Treated Samples**

Sample	Adhesive	Notes	Bond Strength (lbs.)
O	10% Gelatin & 10% Glycerin in Water	Instron test – half of the sample exhibited a break located within plaster layer, while the other half experienced a break at the interface between substrate and plaster. Crumble fracture.	186
H	5% Rhoplex E-330 in Water	Instron test – half of the sample exhibited a break located within plaster layer, while the other half experienced a break at the interface between substrate and plaster.	72
Q	5% Rhoplex E-330 in Water	Instron test – break located at interface between substrate and plaster.	60
Y	5% Rhoplex E-330 in Water	Instron test – break located at the interface between substrate and plaster.	55
I	10% Rhoplex E-330 in Water	Instron test – break located at the interface between substrate and plaster.	46
R	10% Rhoplex E-330 in Water	Instron test – break located at the interface between substrate and plaster.	138
AB	10% Rhoplex E-330 in Water	Instron test – break located at the interface between substrate and plaster.	100
B	5% Rhoplex MC-1834 in Water	Instron test – half of the sample exhibited a break located within plaster layer, while the other half experienced a break at the interface between substrate and plaster.	261
T	5% Rhoplex MC-1834 in Water	Instron test – break located within plaster layer, at the interface between substrate and plaster.	281
L	5% Rhoplex MC-1834 in Water	Instron test – break located at the interface between substrate and plaster.	55
C	10% Rhoplex MC-1834 in Water	Instron test – break located within plaster layer, above the interface between substrate and plaster. Crumbled fracture.	621
K	10% Rhoplex MC-1834 in Water	Instron test – break located at the interface between substrate and plaster.	641
U	10% Rhoplex MC-1834 in Water	Instron test – break located within plaster layer, above the interface between substrate and plaster. Crumbled fracture.	481

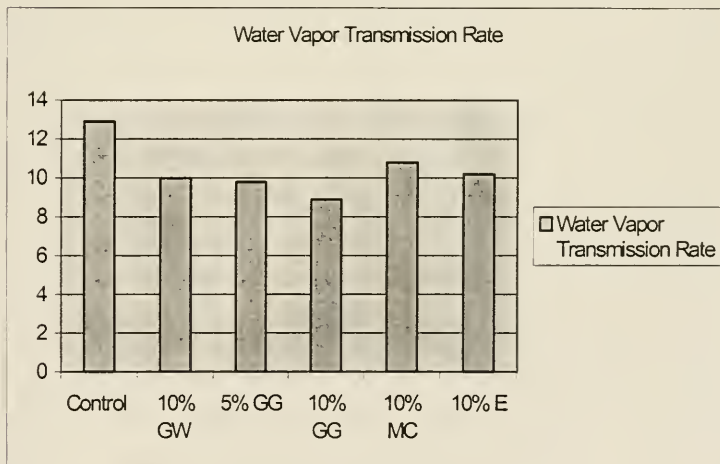


## APPENDIX I: Adhesive Bond Strength of Treated Samples

### Gravimetric Comparison of Water Vapor Transmission for 10% Formulations



### Comparison of Mean Water Vapor Transmission Rates





**APPENDIX J:**  
**Water Vapor Transmission Rate of Treated Samples**





# APPENDIX J: Water Vapor Transmission Rate of Treated Samples

Sample Number		Control-1	Control-2	Control-3	10% GW-1	10% GW-2	10% GW-3
36 hours	Temp	70	70	70	70	70	70
	% RH	75%	75%	75%	75%	75%	75%
	Mass (g)	279.3	283.1	280.0	285.4	290.4	280.6
60 hours	Temp	70	70	70	70	70	70
	RH	35%	35%	35%	35%	35%	35%
	Mass (g)	278.7	281.3	278.2	283.2	287.5	278.8
84 hours	Temp	70	70	70	70	70	70
	RH	48%	48%	48%	48%	48%	48%
	Mass (g)	277.0	278.3	276.6	281.9	286.8	276.8
108 hours	Temp	70	70	70	70	70	70
	RH	50%	50%	50%	50%	50%	50%
	Mass (g)	275.5	276.0	274.9	281.0	285.1	275.4
132 hours	Temp	70	70	70	70	70	70
	RH	35%	35%	35%	35%	35%	35%
	Mass (g)	273.2	274.2	272.0	279.4	283.3	273.5
156 hours	Temp	70	70	70	70	70	70
	RH	65%	65%	65%	65%	65%	65%
	Mass (g)	272.2	270.9	270.5	278.4	282.1	272.8
180 hours	Temp	70	70	70	70	70	70
	RH	42%	42%	42%	42%	42%	42%
	Mass (g)	270.3	268.8	268.5	276.5	280.4	271.1
205 hours	Temp	68	68	68	68	68	68
	RH	35%	35%	35%	35%	35%	35%
	Mass (g)	268.8	267.1	266.8	275.1	278.9	269.7



# APPENDIX J: Water Vapor Transmission Rate of Treated Samples

Sample Number		Control-1	Control-2	Control-3	10% GW-1	10% GW-2	10% GW-3
229 hours	Temp	68	68	68	68	68	68
	RH	50%	50%	50%	50%	50%	50%
	Mass (g)	265.6	263.6	263.7	273.8	277.6	268.3
257 hours	Temp	70	70	70	70	70	70
	RH	56%	56%	56%	56%	56%	56%
	Mass (g)	263.8	260.0	261.7	272.4	276.0	267.2
421 hours	Temp	66	66	66	66	66	66
	RH	38%	38%	38%	38%	38%	38%
	Mass (g)	255.5	252.6	254.3	265.3	269.4	259.9
448 hours	Temp	70	70	70	70	70	70
	% RH	38%	38%	38%	38%	38%	38%
	Mass (g)	254	250.9	252.4	263.9	267.9	258.6
472 hours	Temp	71	71	71	71	71	71
	% RH	41%	41%	41%	41%	41%	41%
	Mass (g)	252.4	250.6	251.1	262.4	266.7	257.3
500 hours	Temp	68	68	68	68	68	68
	% RH	38%	38%	38%	38%	38%	38%
	Mass (g)	251	248	249.7	261.1	265.3	255.9
520 hours	Temp	70	70	70	70	70	70
	% RH	39%	39%	39%	39%	39%	39%
	Mass (g)	249.8	246.8	248.5	260	264.4	254.7
548 hours	Temp	68	68	68	68	68	68
	% RH	42%	42%	42%	42%	42%	42%
	Mass (g)	248.2	245.4	247	258.9	263.1	253.3



## APPENDIX J: Water Vapor Transmission Rate of Treated Samples

Sample Number		Control-1	Control-2	Control-3	10% GW-1	10% GW-2	10% GW-3
572 hours	Temp	68	68	68	68	68	68
	% RH	42%	42%	42%	42%	42%	42%
	Mass (g)	247	244	245.9	257.6	261.8	252.1

Sample Number		5% GG-1	5% GG-2	5% GG-3	10% GG-1	10% GG-2	10% GG-3
36 hours	Temp	70	70	70	70	70	70
	% RH	75%	75%	75%	75%	75%	75%
	Mass (g)	285.1	290.4	286.6	288.6	278.4	291.0
60 hours	Temp	70	70	70	70	70	70
	RH	35%	35%	35%	35%	35%	35%
	Mass (g)	283.2	288.6	284.9	287.0	276.6	289.6
84 hours	Temp	70	70	70	70	70	70
	RH	48%	48%	48%	48%	48%	48%
	Mass (g)	281.9	286.7	283.5	285.7	274.8	288.2
108 hours	Temp	70	70	70	70	70	70
	RH	50%	50%	50%	50%	50%	50%
	Mass (g)	280.5	284.9	282.4	284.3	273.0	286.8
132 hours	Temp	70	70	70	70	70	70
	RH	35%	35%	35%	35%	35%	35%
	Mass (g)	279.0	283.0	280.5	282.9	271.5	285.4
156 hours	Temp	70	70	70	70	70	70
	RH	65%	65%	65%	65%	65%	65%
	Mass (g)	278.1	281.4	280.0	282.2	270.1	284.7



## APPENDIX J: Water Vapor Transmission Rate of Treated Samples

Sample Number		5%	5%	5%	10%	10%	10%
		GG-1	GG-2	GG-3	GG-1	GG-2	GG-3
180 hours	Temp	70	70	70	70	70	70
	RH	42%	42%	42%	42%	42%	42%
	Mass (g)	276.6	279.9	278.2	280.5	268.4	283.1
205 hours	Temp	68	68	68	68	68	68
	RH	35%	35%	35%	35%	35%	35%
	Mass (g)	275.0	277.9	276.8	279.3	266.9	281.7
229 hours	Temp	68	68	68	68	68	68
	RH	50%	50%	50%	50%	50%	50%
	Mass (g)	273.4	275.7	275.7	278.1	265.4	280.4
257 hours	Temp	70	70	70	70	70	70
	RH	56%	56%	56%	56%	56%	56%
	Mass (g)	272.5	273.6	274.3	276.7	262.0	279.5
421 hours	Temp	66	66	66	66	66	66
	RH	38%	38%	38%	38%	38%	38%
	Mass (g)	266	267.4	267.8	270.2	257.6	272.6
448 hours	Temp	70	70	70	70	70	70
	% RH	38%	38%	38%	38%	38%	38%
	Mass (g)	264.6	266.1	266.5	269	256.2	271.3
472 hours	Temp	71	71	71	71	71	71
	% RH	41%	41%	41%	41%	41%	41%
	Mass (g)	263.4	264.8	265.7	267.7	255	270.2
500 hours	Temp	68	68	68	68	68	68
	% RH	38%	38%	38%	38%	38%	38%
	Mass (g)	262	263.6	264.1	266.4	253.7	269





# APPENDIX J: Water Vapor Transmission Rate of Treated Samples

Sample Number		5% GG-1	5% GG-2	5% GG-3	10% GG-1	10% GG-2	10% GG-3
520 hours	Temp	70	70	70	70	70	70
	% RH	39%	39%	39%	39%	39%	39%
	Mass (g)	261.1	262.6	263.1	265.5	252.8	268
548 hours	Temp	68	68	68	68	68	68
	% RH	42%	42%	42%	42%	42%	42%
	Mass (g)	260	261.4	261.9	264.2	251.5	266.9
572 hours	Temp	68	68	68	68	68	68
	% RH	42%	42%	42%	42%	42%	42%
	Mass (g)	258.8	260.3	261.0	263.1	250.6	265.7

Sample Number		10% MC-1	10% MC-2	10% MC-3	10% E-1	10% E-2	10% E-3
36 hours	Temp	70	70	70	70	70	70
	% RH	75%	75%	75%	75%	75%	75%
	Mass (g)	294.0	301.0	309.0	301.0	308.0	304.8
60 hours	Temp	70	70	70	70	70	70
	RH	35%	35%	35%	35%	35%	35%
	Mass (g)	290.7	298.8	307.4	299.5	307.9	302.9
84 hours	Temp	70	70	70	70	70	70
	RH	48%	48%	48%	48%	48%	48%
	Mass (g)	289.6	297.0	306.1	298.3	306.5	300.6
108 hours	Temp	70	70	70	70	70	70
	RH	50%	50%	50%	50%	50%	50%
	Mass (g)	288.1	294.9	304.7	296.9	304.9	300.2



## APPENDIX J: Water Vapor Transmission Rate of Treated Samples

Sample Number		10% MC-1	10% MC-2	10% MC-3	10% E-1	10% E-2	10% E-3
132 hours	Temp	70	70	70	70	70	70
	RH	35%	35%	35%	35%	35%	35%
	Mass (g)	286.4	293.3	303.3	295.3	303.2	298.6
156 hours	Temp	70	70	70	70	70	70
	RH	65%	65%	65%	65%	65%	65%
	Mass (g)	285.3	291.8	302.6	294.5	301.7	297.7
180 hours	Temp	70	70	70	70	70	70
	RH	42%	42%	42%	42%	42%	42%
	Mass (g)	283.4	290.0	300.8	292.6	300.0	295.9
205 hours	Temp	68	68	68	68	68	68
	RH	35%	35%	35%	35%	35%	35%
	Mass (g)	282.0	288.6	299.4	291.3	298.7	294.4
229 hours	Temp	68	68	68	68	68	68
	RH	50%	50%	50%	50%	50%	50%
	Mass (g)	273.7	286.8	298.1	290.0	297.4	293.3
257 hours	Temp	70	70	70	70	70	70
	RH	56%	56%	56%	56%	56%	56%
	Mass (g)	273.1	285.3	296.8	288.8	295.9	291.9
421 hours	Temp	66	66	66	66	66	66
	RH	38%	38%	38%	38%	38%	38%
	Mass (g)	271.9	278.6	290.2	282	289.8	285.3
448 hours	Temp	70	70	70	70	70	70
	% RH	38%	38%	38%	38%	38%	38%
	Mass (g)	270.5	277.1	288.9	280.7	288.4	283.8



## APPENDIX J: Water Vapor Transmission Rate of Treated Samples

Sample Number		10% MC-1	10% MC-2	10% MC-3	10% E-1	10% E-2	10% E-3
472 hours	Temp	71	71	71	71	71	71
	% RH	41%	41%	41%	41%	41%	41%
	Mass (g)	269.4	275.8	287.5	279.6	287.2	282.6
500 hours	Temp	68	68	68	68	68	68
	% RH	38%	38%	38%	38%	38%	38%
	Mass (g)	268	274.5	286.2	278.2	285.9	281.1
520 hours	Temp	70	70	70	70	70	70
	% RH	39%	39%	39%	39%	39%	39%
	Mass (g)	267.1	273.5	285.1	277.2	285	280.2
548 hours	Temp	68	68	68	68	68	68
	% RH	42%	42%	42%	42%	42%	42%
	Mass (g)	265.7	272.1	283.9	275.8	283.7	279.1
572 hours	Temp	68	68	68	68	68	68
	% RH	42%	42%	42%	42%	42%	42%
	Mass (g)	264.6	270.9	282.8	274.6	282.7	278



## APPENDIX J: Water Vapor Transmission Rate of Treated Samples

Summary of Gravimetric Loss by Water Vapor Transmission

Sample Solution	Container Weight (grams)	Sample Weight (grams)	Original Weight (grams)
Control-1	5.4	98.2	281.6
Control-2	4.8	99.2	285.3
Control-3	5.6	94.6	281.0
10% GW-1 (10% Gelatin in water)	5.5	100.6	284.6
10% GW-2 (10% Gelatin in water)	5.4	109.6	292.1
10% GW-3 (10% Gelatin in water)	5.6	93.8	280.9
5% GG-1 (5% Gelatin with 10% Glycerin in water)	5.5	101.4	284.4
5% GG-2 (5% Gelatin with 10% Glycerin in water)	5.2	108.4	292.0
5% GG-3 (5% Gelatin with 10% Glycerin in water)	5.0	105.9	287.5
10% GG-1 (10% Gelatin with 10% Glycerin in water)	5.0	105.8	284.3
10% GG-2 (10% Gelatin with 10% Glycerin in water)	5.1	99.4	279.8
10% GG-3 (10% Gelatin with 10% Glycerin in water)	5.4	107.0	291.7
10% MC-1 (10% MC-1834 in water)	5.5	121.0	298.1
10% MC-2 (10% MC-1834 in water)	5.1	120.7	302.7
10% MC-3 (10% MC-1834 in water)	5.3	124.8	309.7
10% E-1 (10% E-330 in water)	5.5	117.6	299.5
10% E-2 (10% E-330 in water)	5.6	123.6	310.7
10% E-3 (10% E-330 in water)	5.2	121.6	305.7





## **APPENDIX K:**

### **Accelerated Weathering – Preliminary Testing**





New Orleans  
Brick - Yellow



New Orleans  
Brick - Red



Modern Brick -  
University of PA

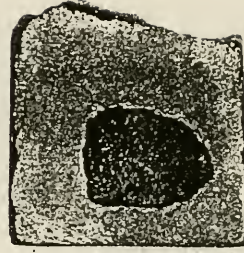


Modern Brick -  
Extruded

321



Mesa Verde  
Sandstone



Sample X Blister  
Mesa Verde Sandstone

## Accelerated Weathering - Preliminary Testing



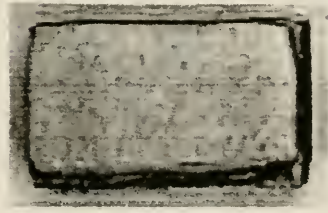
**APPENDIX L:**

**Substrate: Modern Extruded Brick**

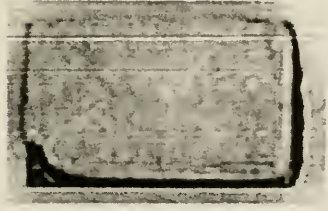
**Surface Finishes: Reddish Brown Plaster with a White Wash**

**Weathered Samples – Not Treated**

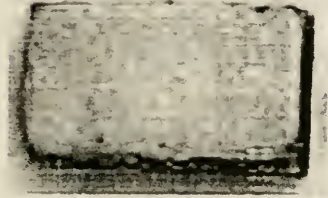




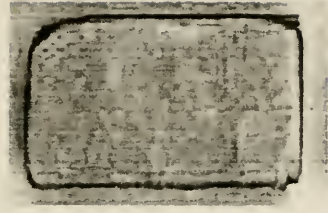
Sample A



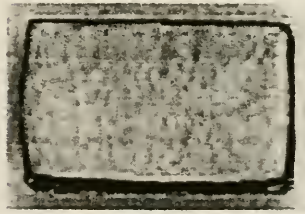
Sample B



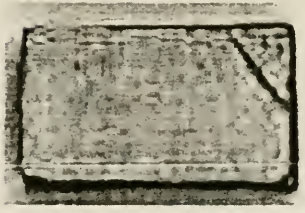
Sample C



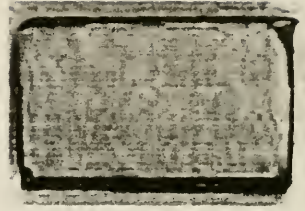
Sample D



Sample E



Sample F



Sample G

## Weathered Samples - Not Treated





**APPENDIX M:**

**Substrate: Modern Extruded Brick**

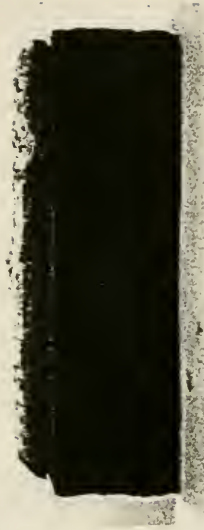
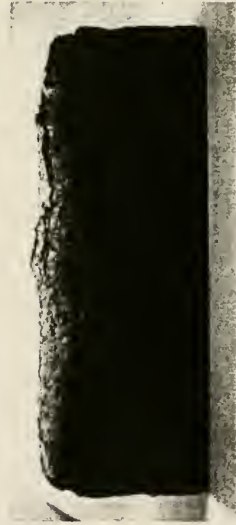
**Surface Finishes: Reddish Brown Plaster with a White Wash**

**Weathered Samples – Treated**

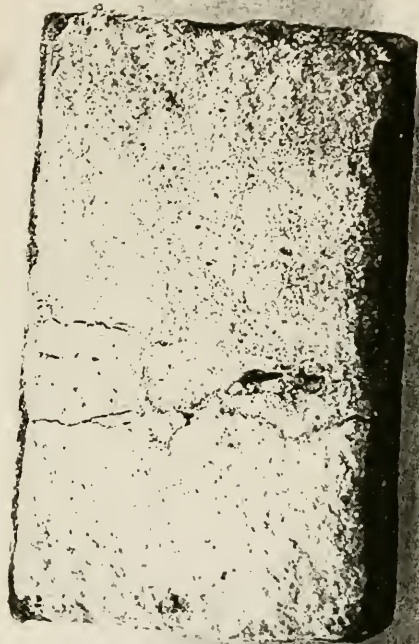




Sample H - Treated with 10% Gelatin with 10% Glycerin

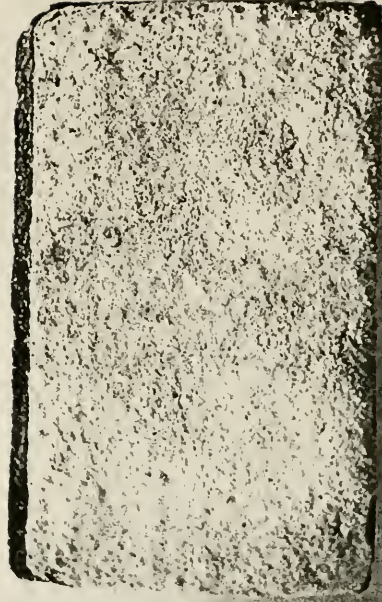






Sample I - Treated with 10% Gelatin with 10% Glycerin



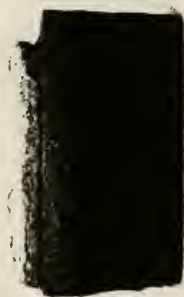
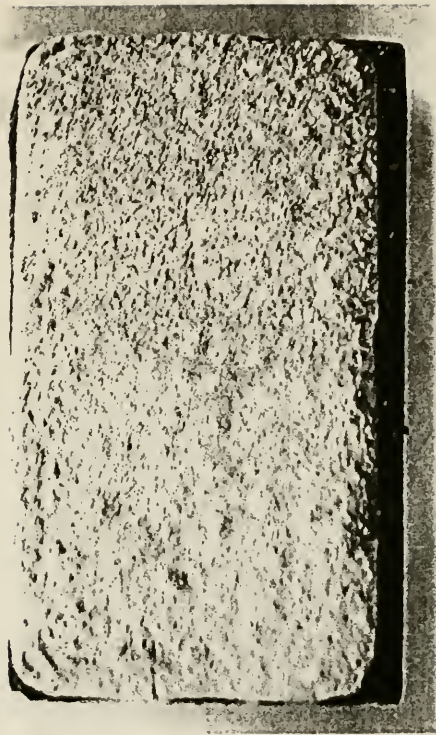


Sample J - Treated with 10% Gelatin with 10% Glycerin

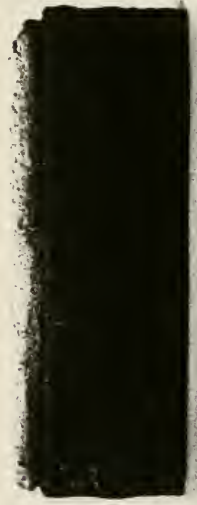




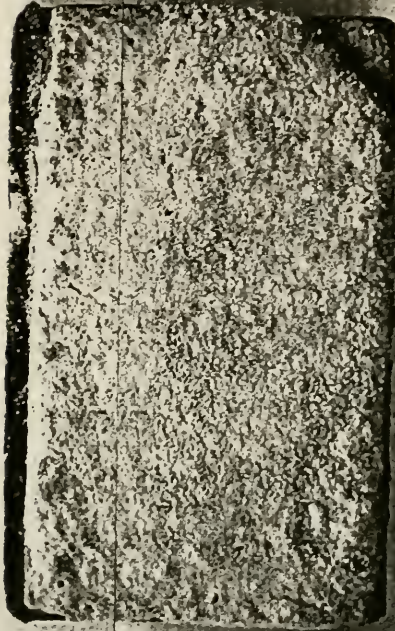




Sample K - Treated with 10% Rhoplex E-330



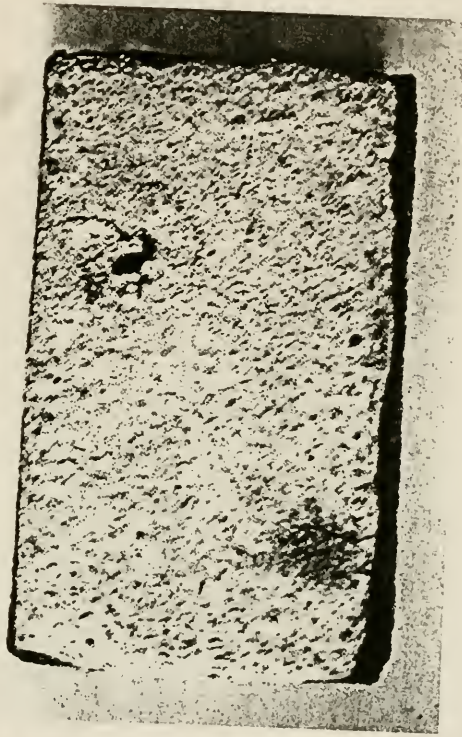




Sample L - Treated with 10% Rhoplex E-330





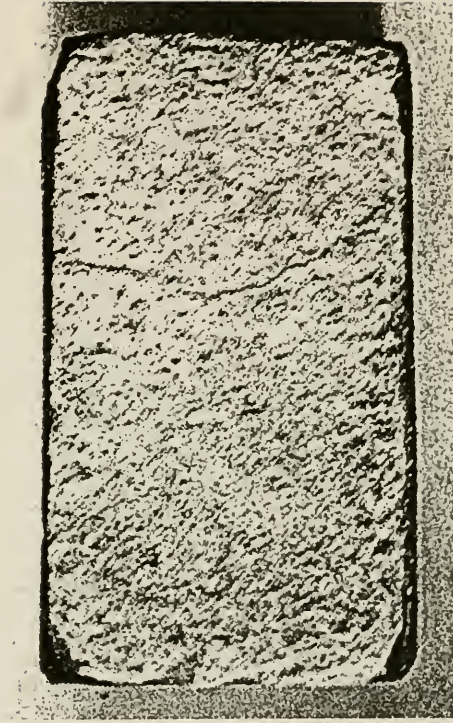


Sample M - Treated with 10% Rhoplex E-330

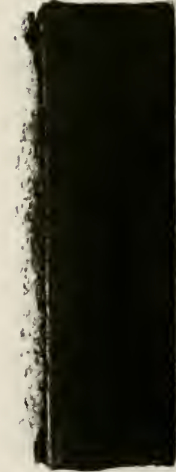






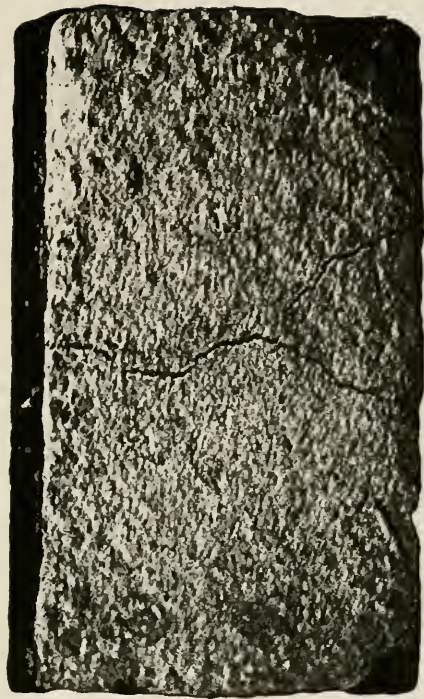


Sample N - Treated with 10% Rhoplex MC-1834





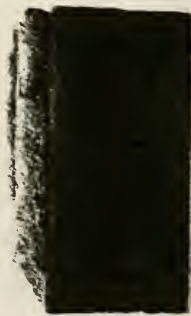
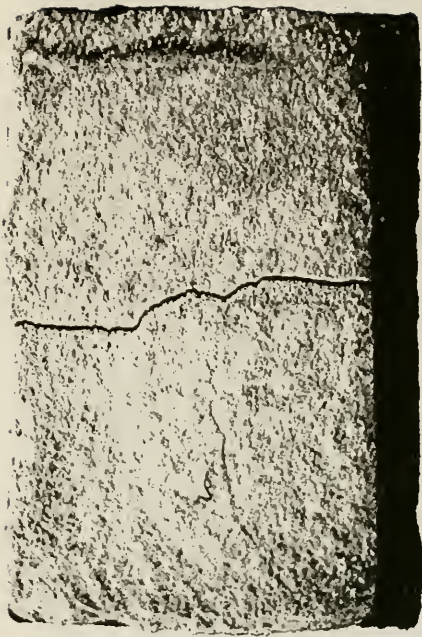




Sample O - Treated with 10% Rhoplex MC-1834



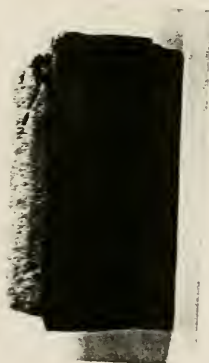
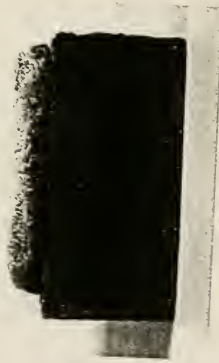




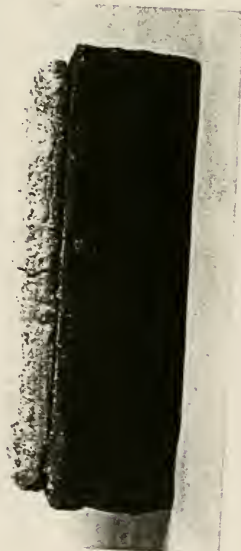
Sample P - Treated with 10% Rhoplex MC-1834





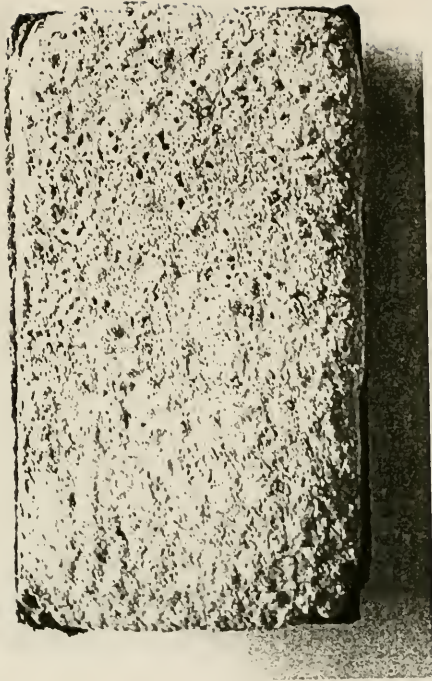


Sample Q - Treated with 5% Gelatin





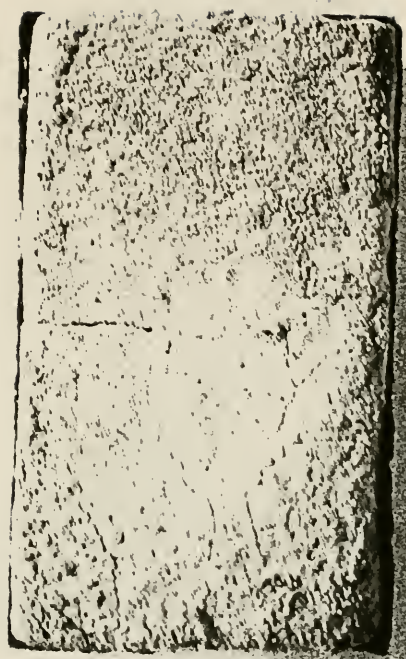




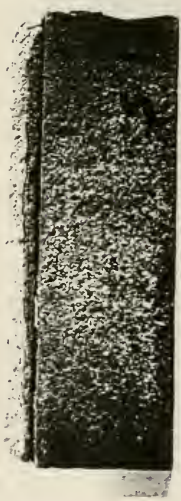
Sample R - Treated with 5% Gelatin



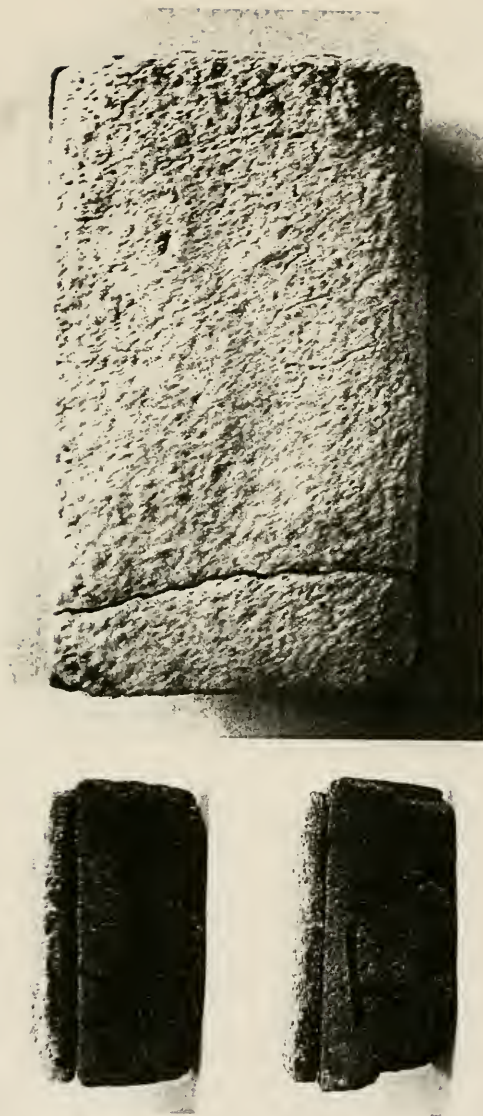




Sample S - Treated with 5% Gelatin



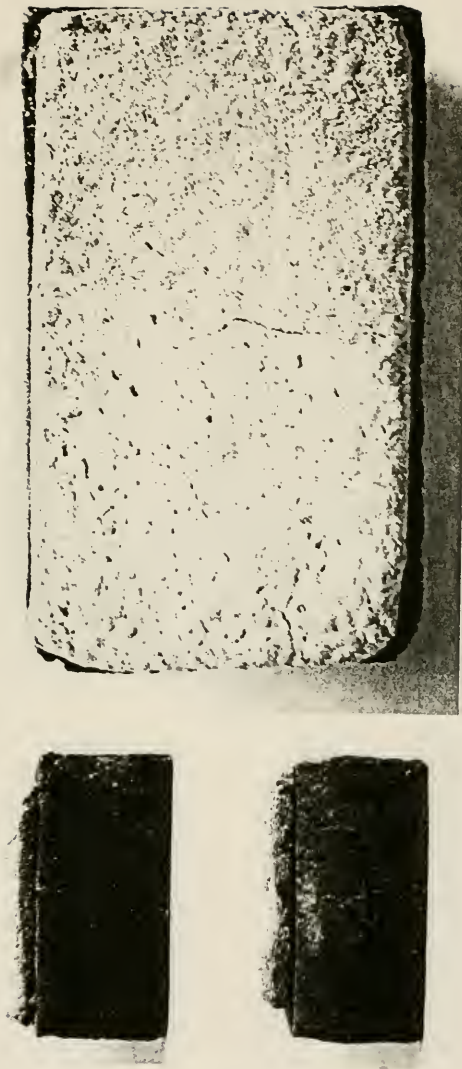




Sample T - Treated with 5% Gelatin with 10% Glycerin



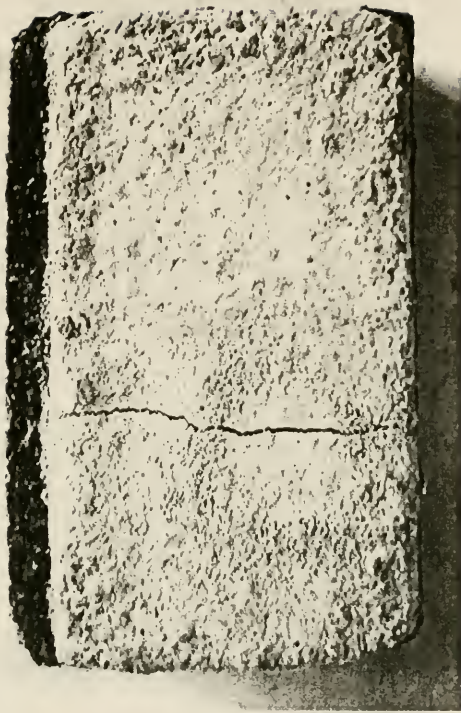




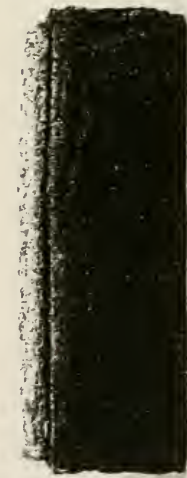
Sample U - Treated with 5% Gelatin with 10% Glycerin





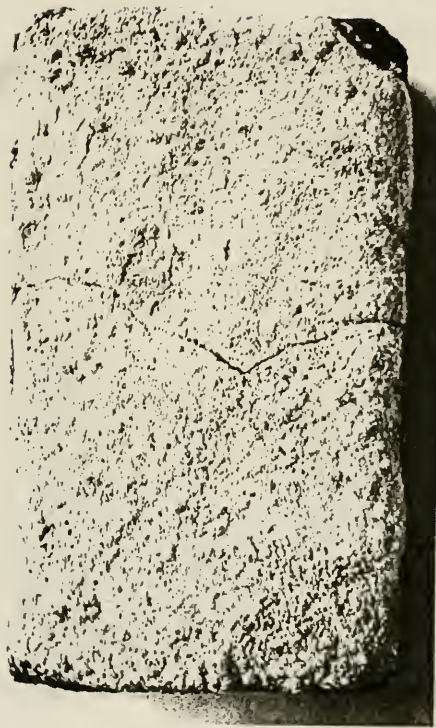


Sample V - Treated with 5% Gelatin with 10% Glycerin





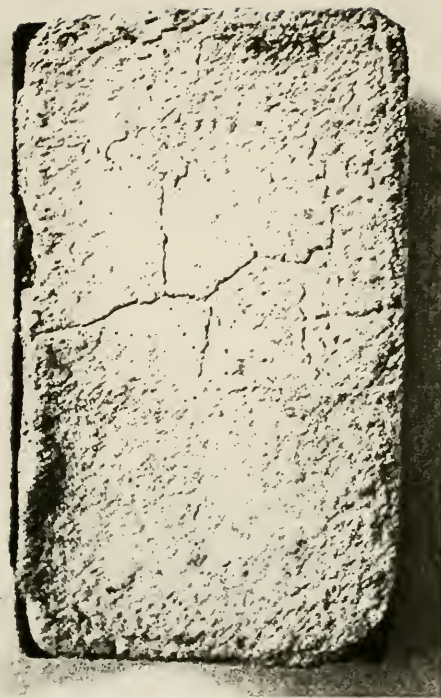




Sample W - Treated with 10% Gelatin



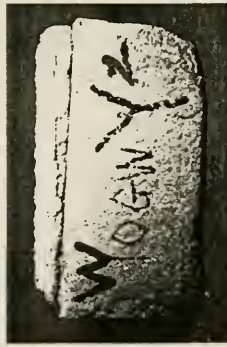




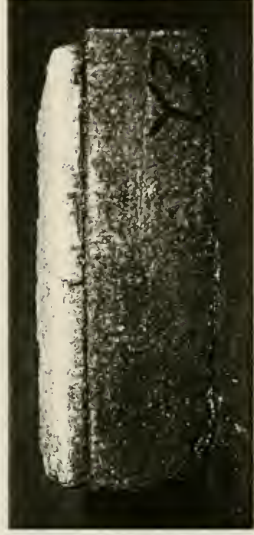
Sample X - Treated with 10% Gelatin





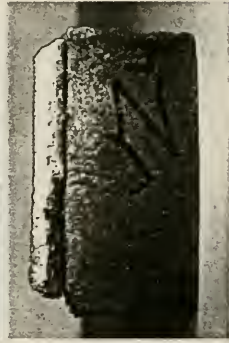


Sample Y - Treated with 10% Gelatin







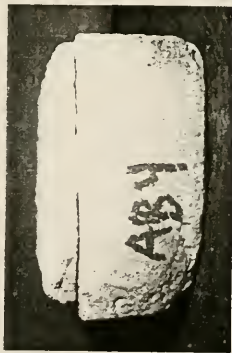
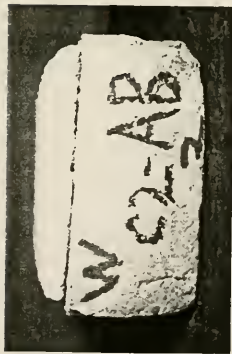


Sample Z - Treated with Water

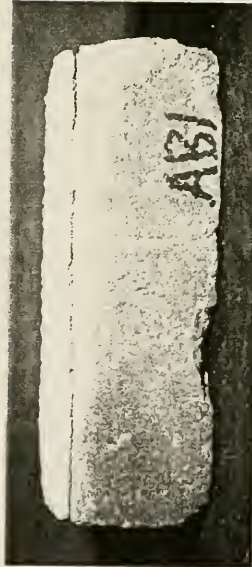








Sample AB - Treated with Water





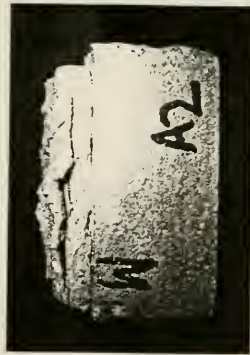
## **APPENDIX N:**

**Substrate: Modern Extruded Brick**

**Surface Finishes: Reddish Brown Plaster with a Reddish Brown Wash**

**Weathered Samples – Treated**





Sample A - Treated with Water

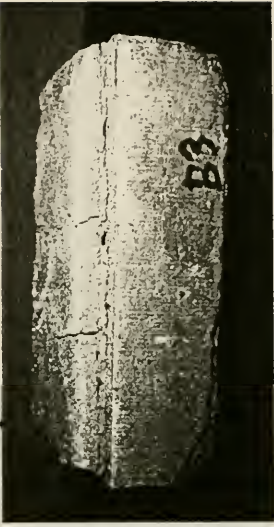






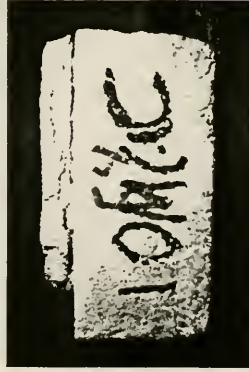


Sample B - Treated with 5% Phoplex MC-1834

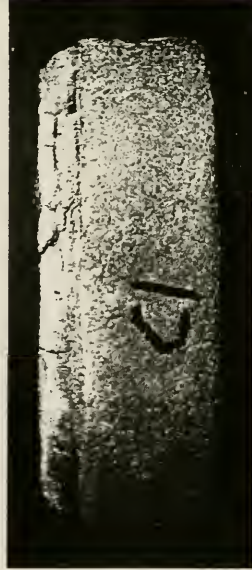








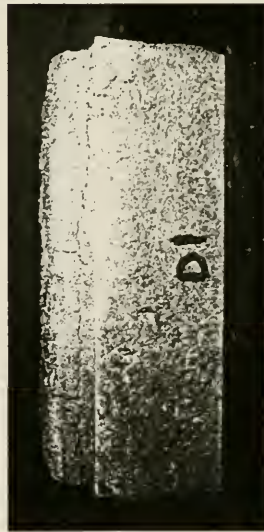
Sample C - Treated with 10% Phoplex MC-1834





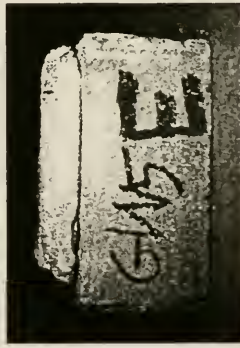


Sample D - Treated with 5% Gelatin & %10 Glycerin

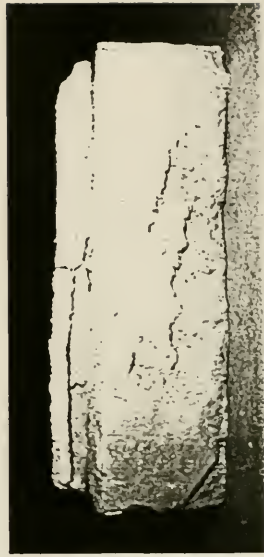




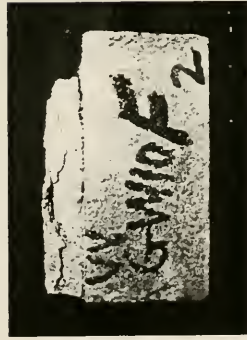




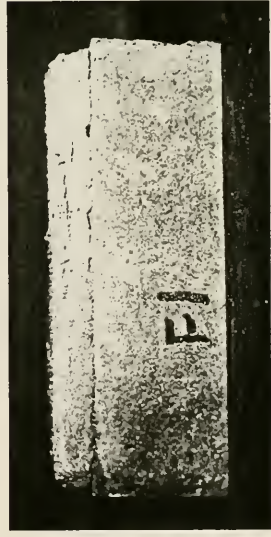
Sample E - Treated with 5% Gelatin





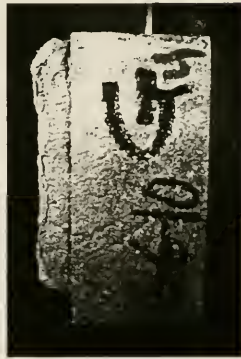


Sample F - Treated with 10% Gelatin

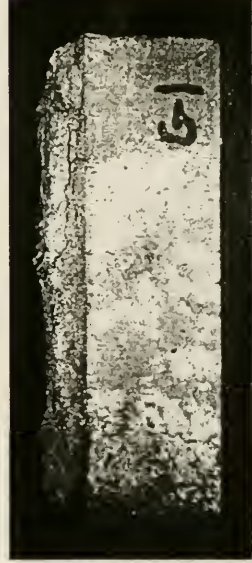




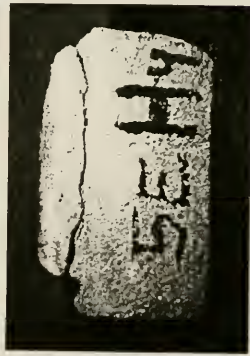




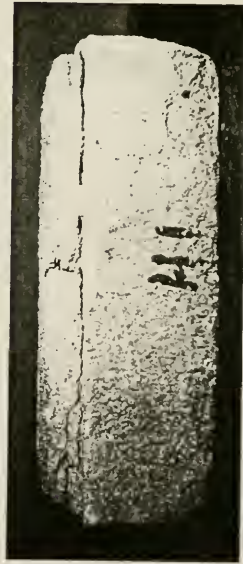
Sample G - Treated with 10% Gelatin & 10% Glycerin





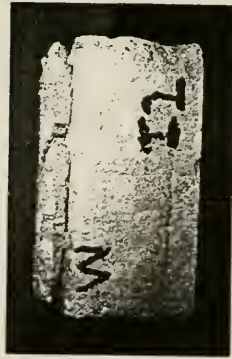


Sample H - Treated with 5% Rhoplex E-330

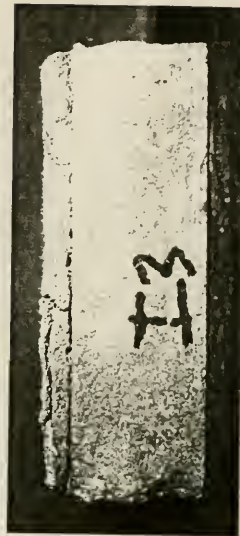
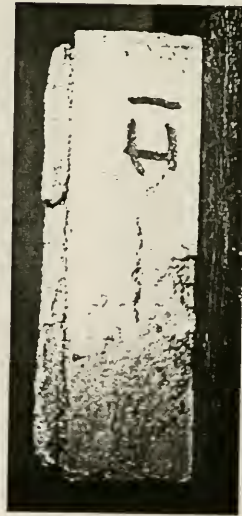




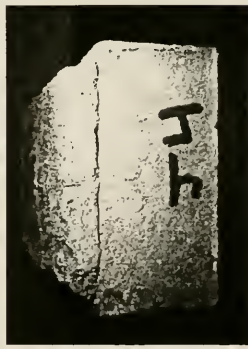
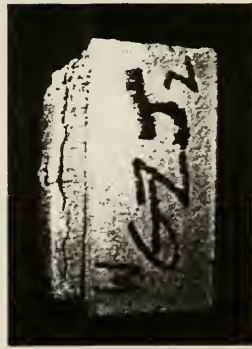




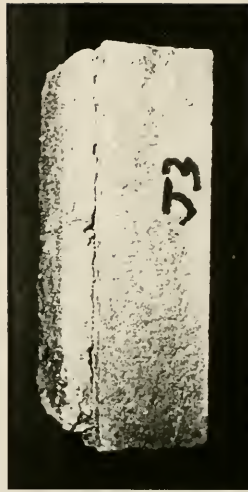
Sample I - Treated with 10% Rhoplex E-330





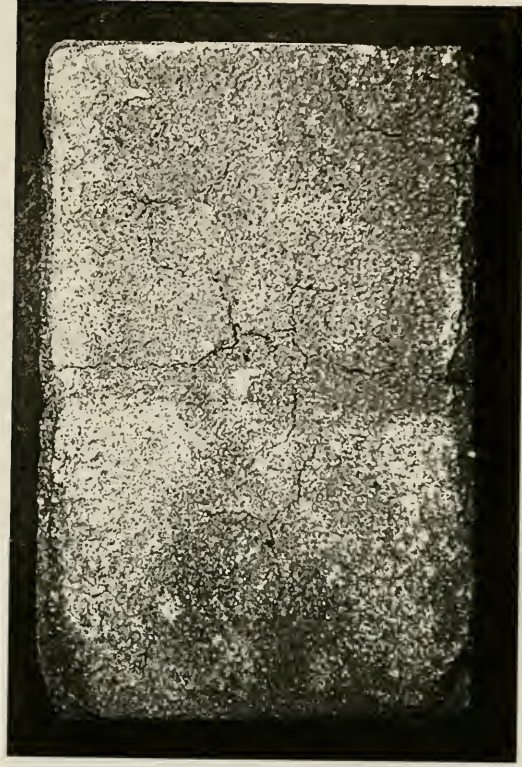
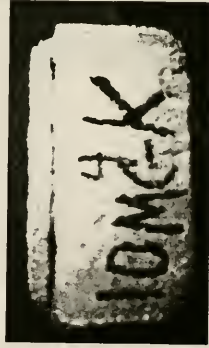


Sample J - Treated with Water

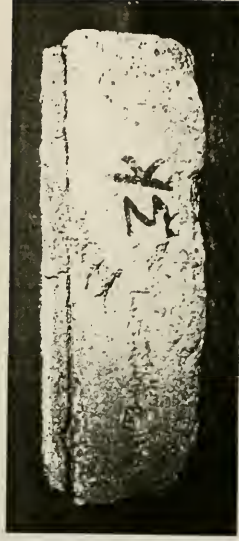
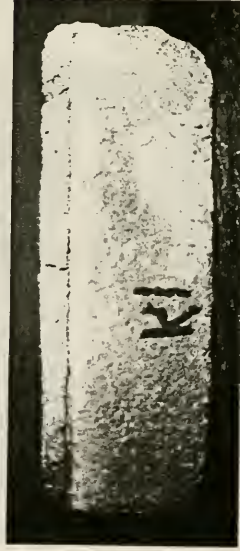




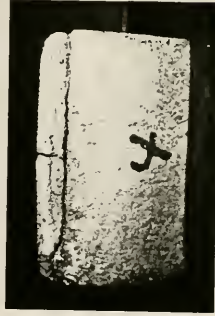




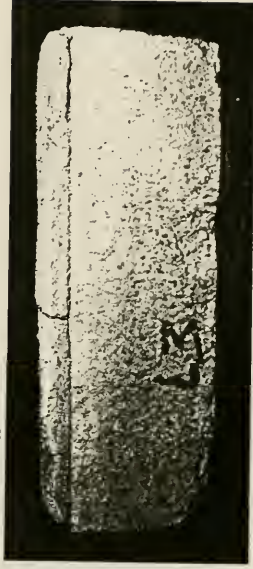
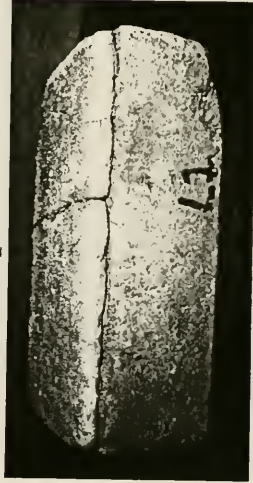
Sample K - Treated with 10% Rhoplex MC-1834





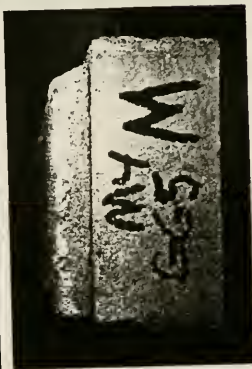
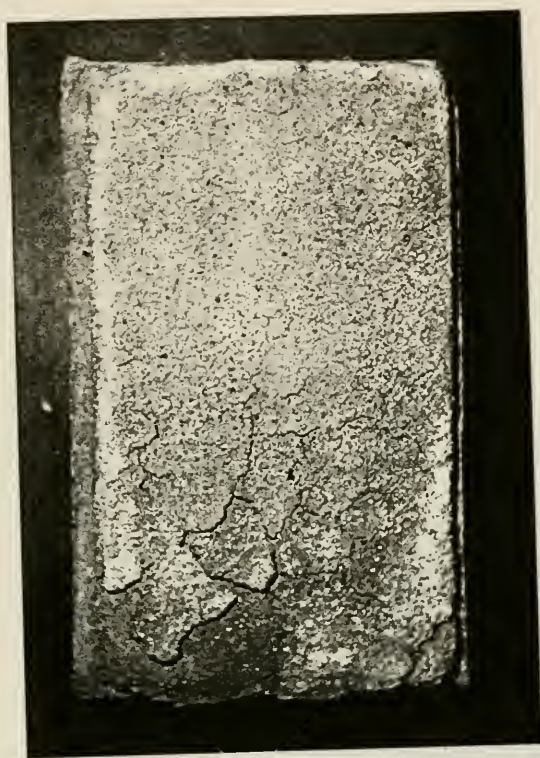


Sample L - Treated with 5% Rhoplex MC-1834

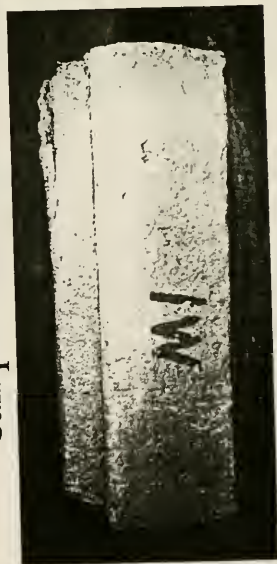
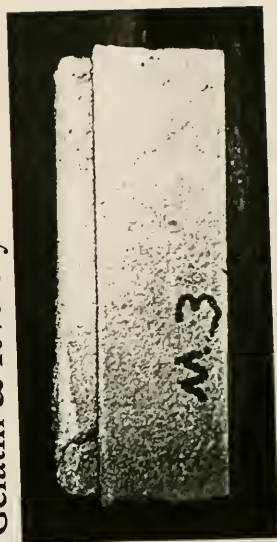








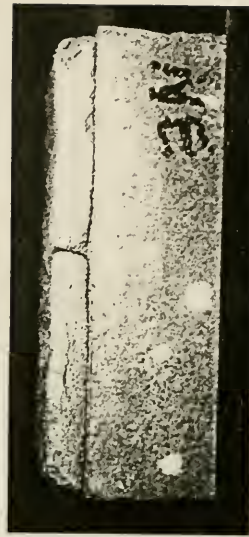
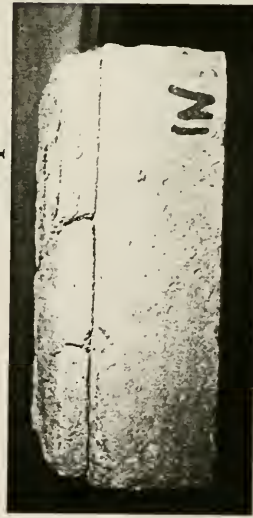
Sample M - Treated with 5% Gelatin & 10% Glycerin





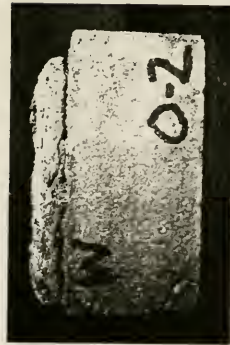


Sample N - Treated with 5% Gelatin

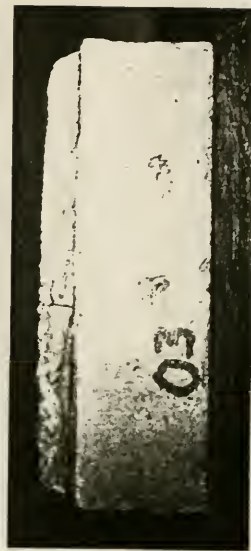
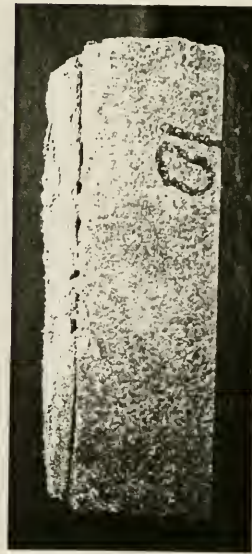




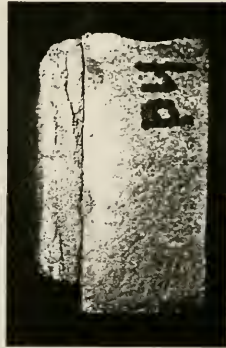




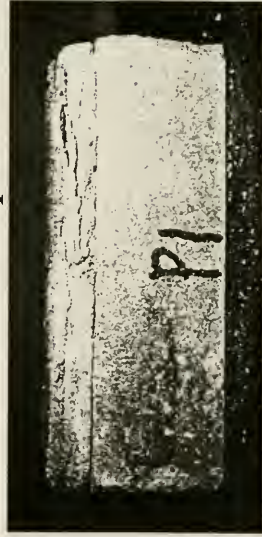
Sample O - Treated with 5% Rhoplex E-330





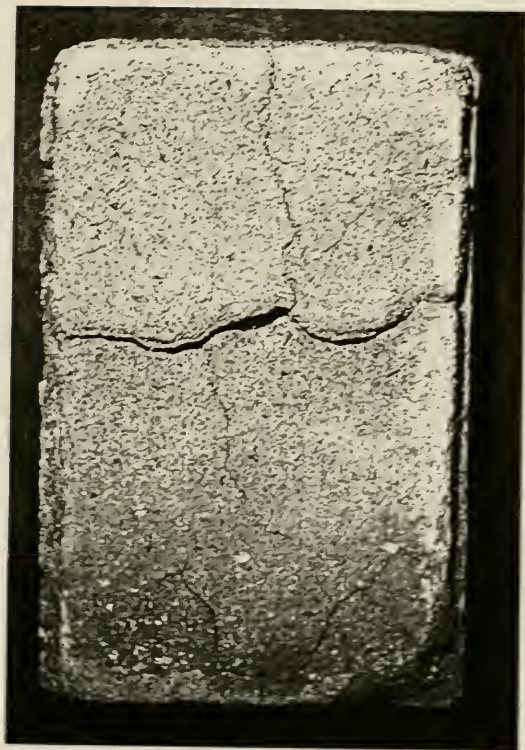
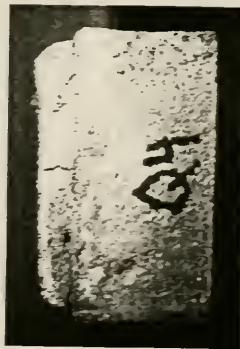
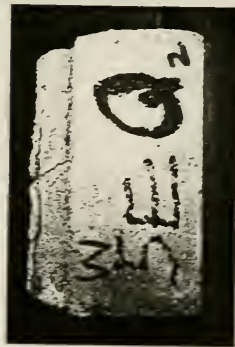


Sample P - Treated with 10% Gelatin

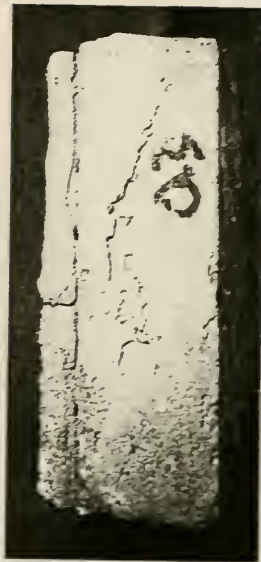
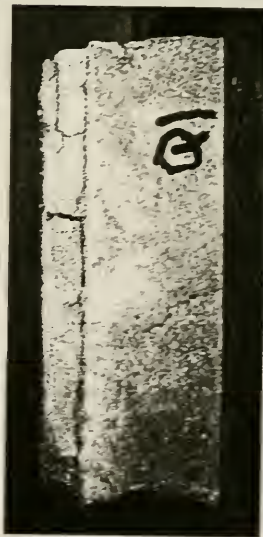




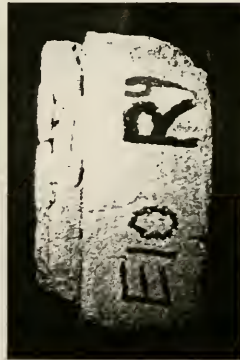




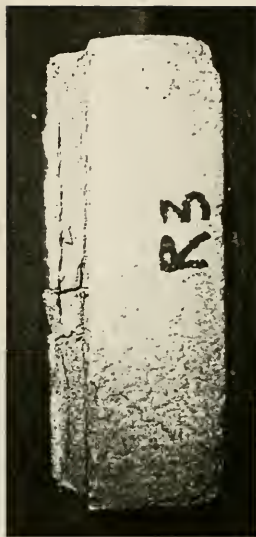
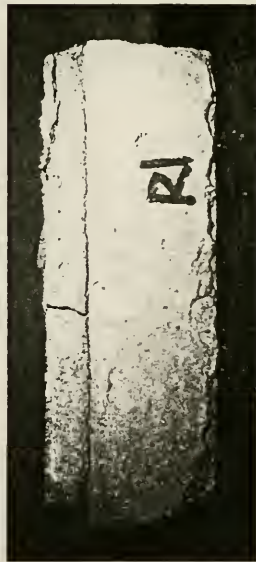
Sample Q - Treated with 5% Rhoplex E-330





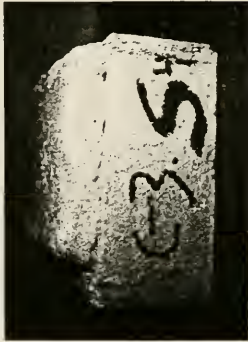
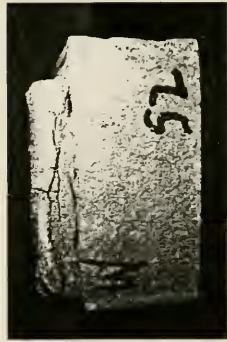


Sample R - Treated with 10% Rhoplex E-330

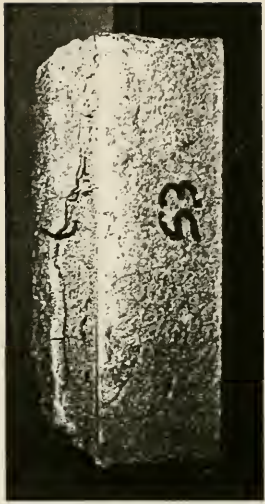
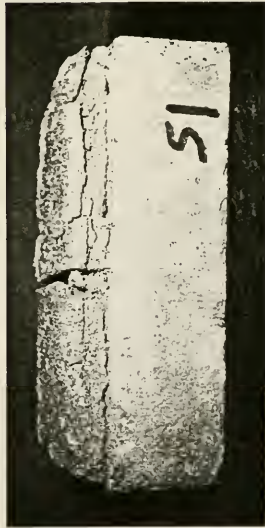




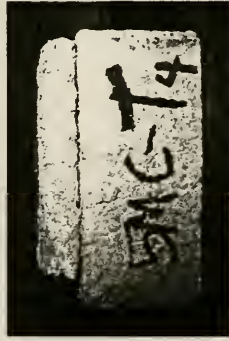




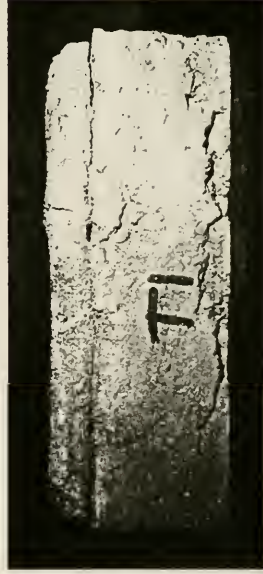
Sample S - Treated with Water





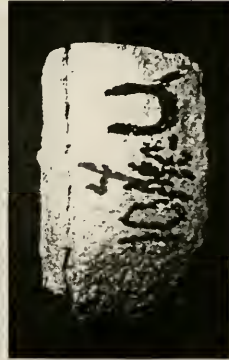
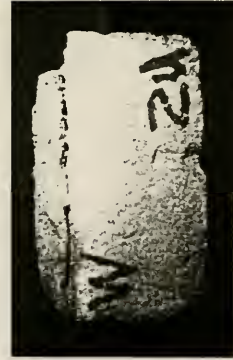


Sample T - Treated with 5% Rhoplex MC-1834

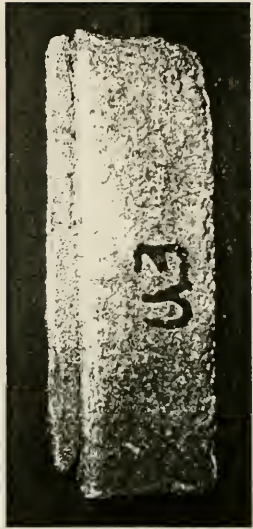
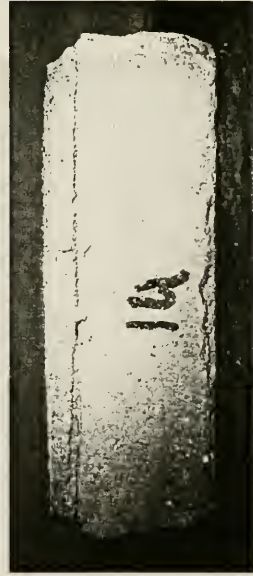




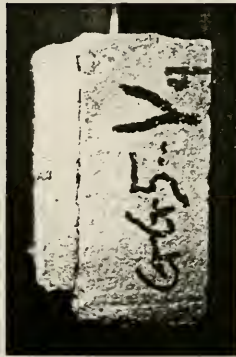




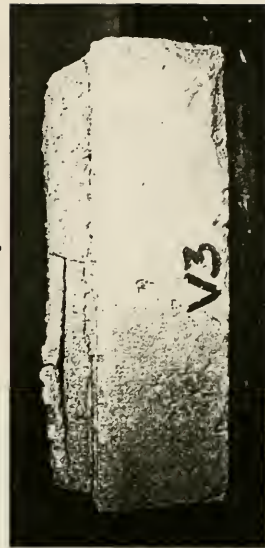
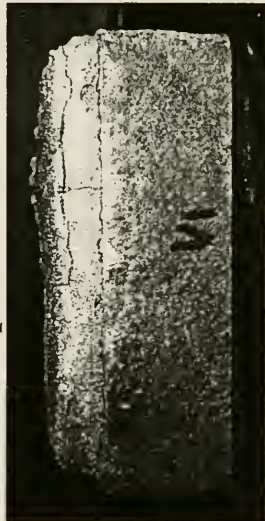
Sample U - Treated with 10% Rhoplex MC-1834





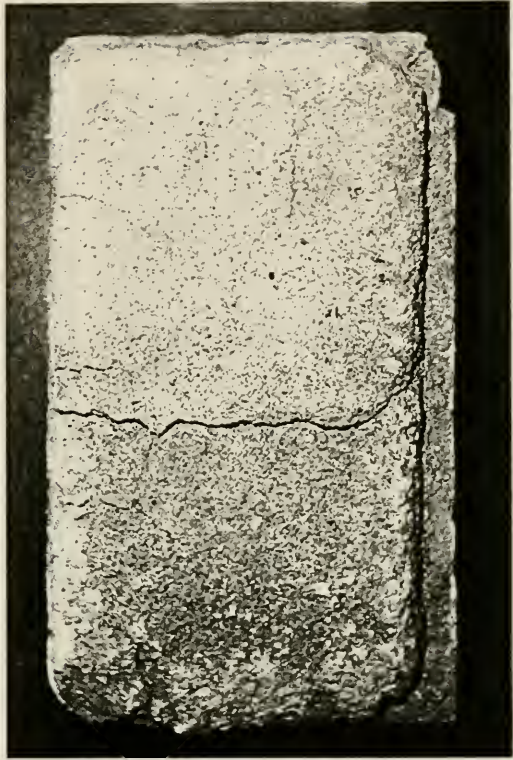


Sample V - Treated with 5% Gelatin & 10% Glycerin

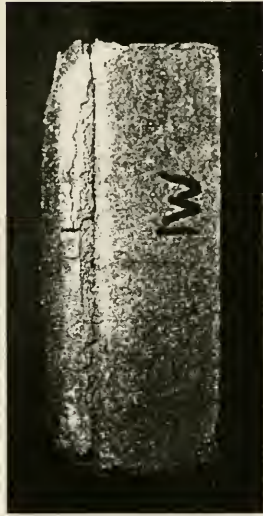




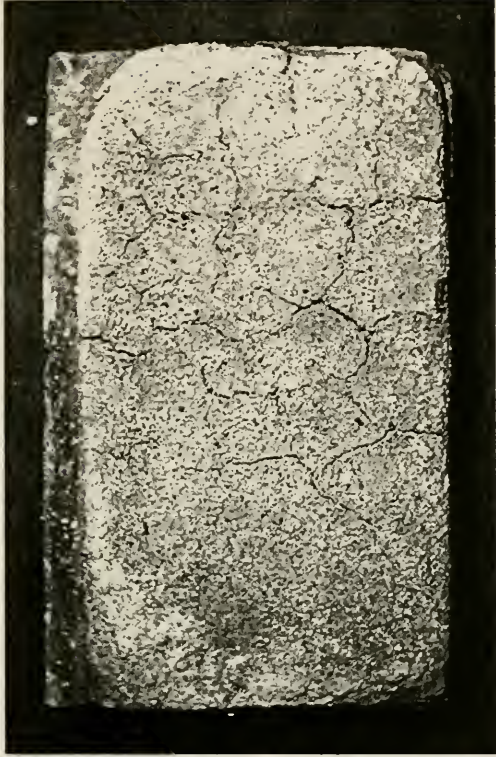




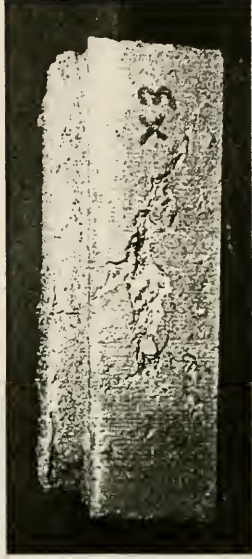
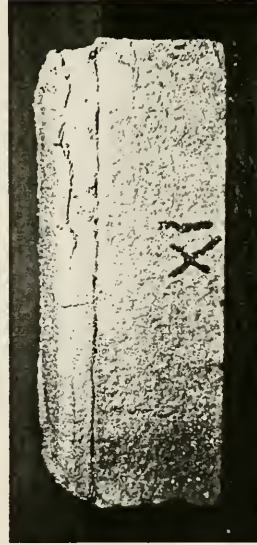
Sample W - Treated with 5% Gelatin







Sample X - Treated with 10% Gelatin

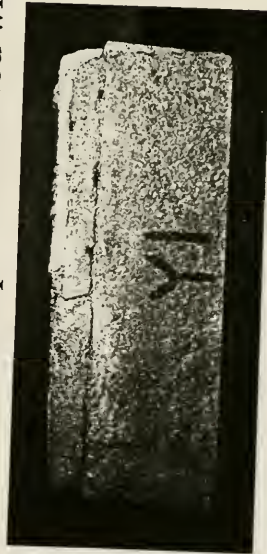




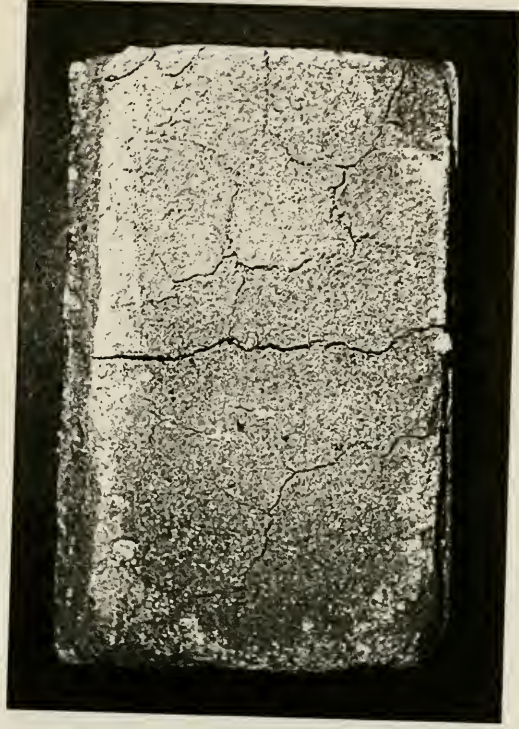
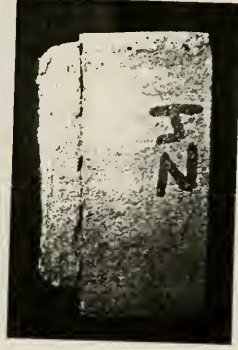




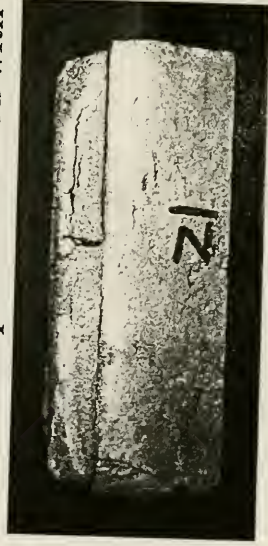
Sample Y - Treated with 5% Rhoplex E-330





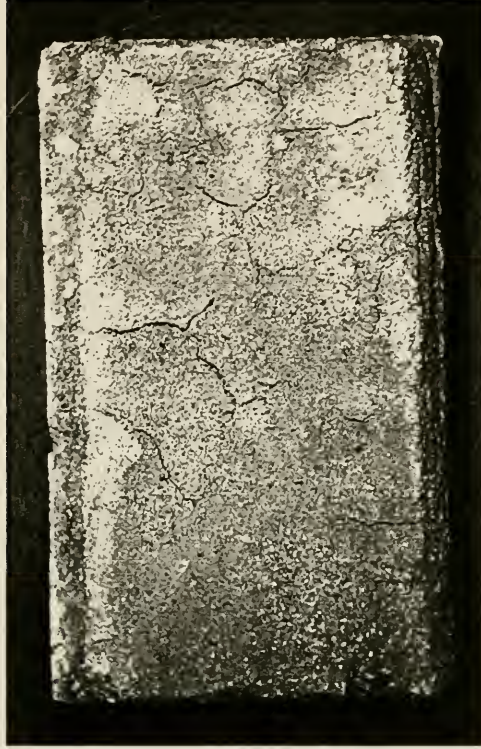
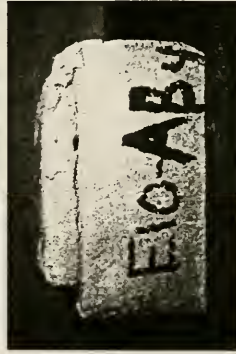


Sample Z - Treated with 10% Gelatin & 10% Glycerin









Sample AB - Treated with 10% Rhoplex E-330





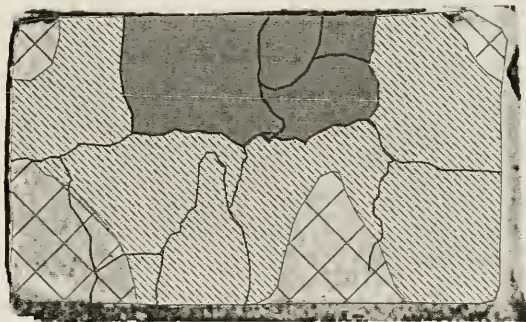
## **APPENDIX O:**

**Substrate: Modern Extruded Brick (Penn)**

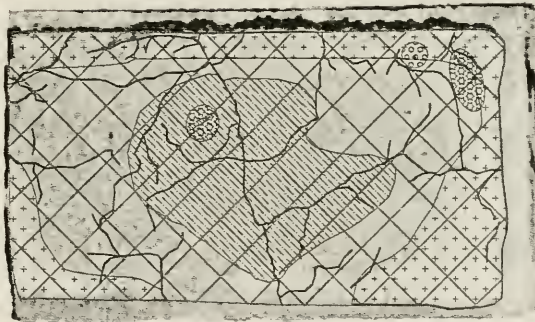
**Surface Finishes: Reddish Brown Plaster with a Reddish Brown Wash**

**Weathered Samples – Digital Condition Maps**





Sample A:  
Treated with 10 cc. Water  
Follow-up Treatment: 12 cc. Water



Sample J:  
Treated with 11 cc. Water

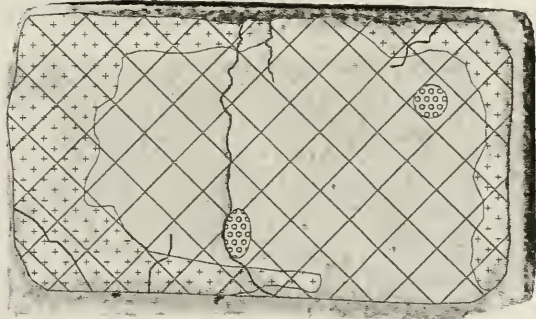


Sample S:  
Treated with 11 cc. Water

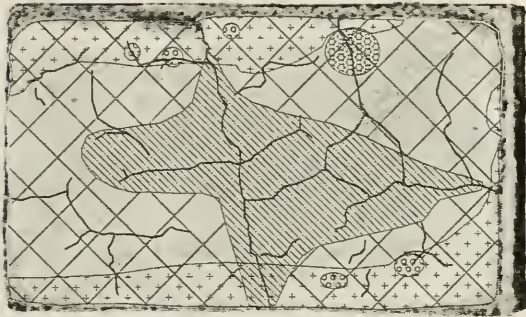
# KEY

	Plaster Outline		Blister		Water Damage
	Cracks		Interfacial Delamination		Loss
	Salts		Substrate Detachment		Treated Area

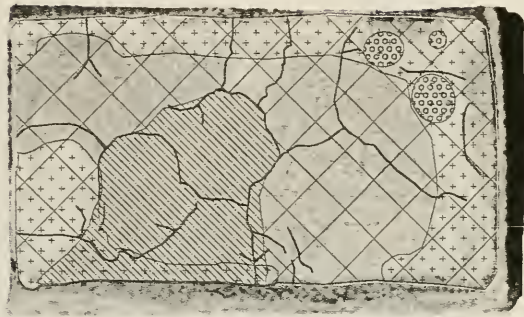




Sample Vi:  
Treated with 10 cc. 5% Gelatin in  
Water



Sample Ni:  
Treated with 9 cc. 5% Gelatin in  
Water



Sample Ei:  
Treated with 8 cc. 5% Gelatin in  
Water

# KEY

	Plaster Outline		Blisters		Water Damage
	Cracks		Interface Delamination		Loss
	Salts		Substrate Detachment		Treated Area





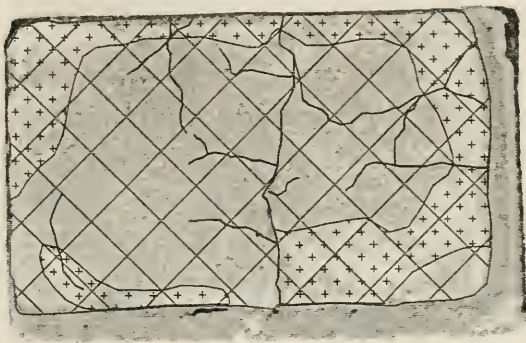




Sample Mi:  
Treated with 6 cc. 5% Gelatin & 10%  
Glycerin in Water



Sample Di:  
Treated with 9 cc. 5% Gelatin & 10%  
Glycerin in Water



Sample Vi:  
Treated with 7 cc. 5% Gelatin & 10%  
Glycerin in Water

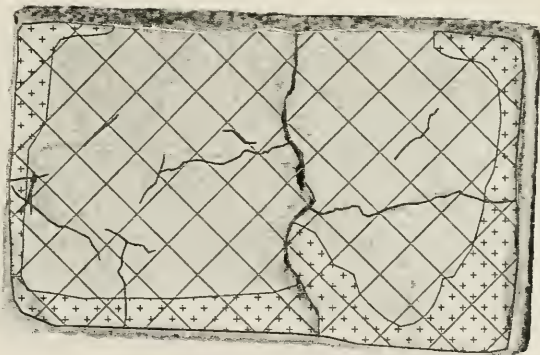
# KEY

	Plaster Outline		Blisters		Water Damage
	Cracks		Interfacial Delamination		Loss
	Salts		Substrate Detachment		Treated Area

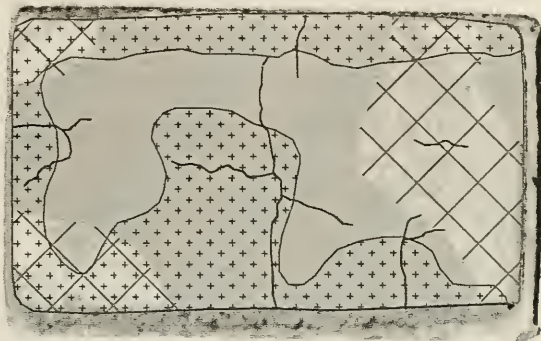




Sample H1  
Treated with 6 cc. 5% E-330 in  
Water



Sample Q1  
Treated with 9 cc. 5% E-330 in  
Water

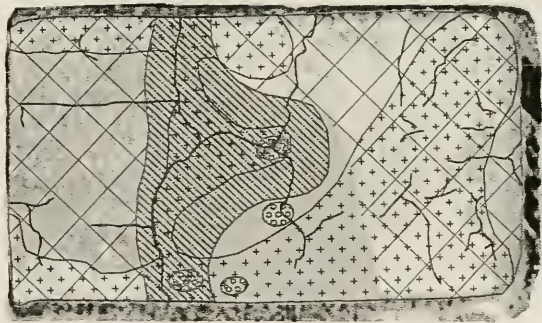


Sample Y1  
Treated with 13 cc. 5% E-330 in  
Water

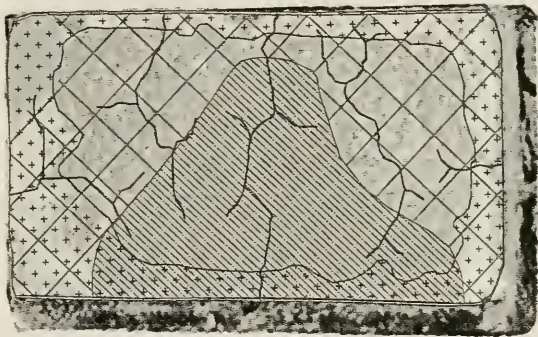
# KEY

	Plaster Outline		Blisters		Water Damage
	Cracks		Interfacial Delamination		Loss
	Salts		Substrate Detachment		Treated Area

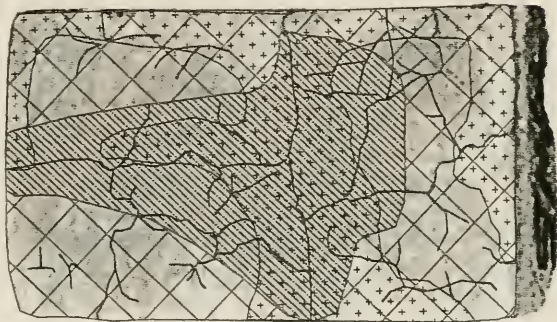




Sample B:  
Treated with 9 cc. 5% MC-1834  
in water

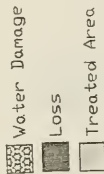
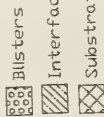
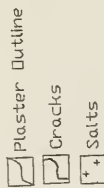


Sample L:  
Treated with 9 cc. 5% MC-1834 in  
water



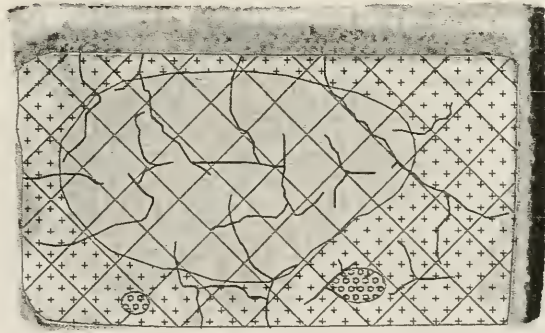
Sample L:  
Treated with 7 cc. 5% MC-1834  
in water

# KEY

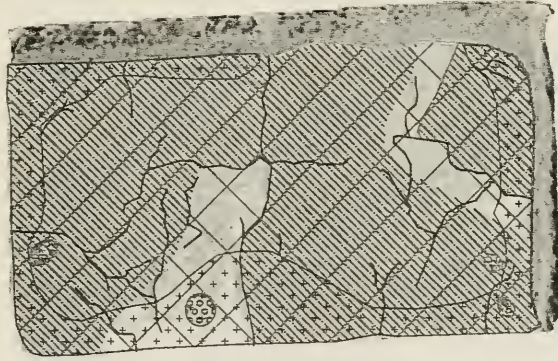




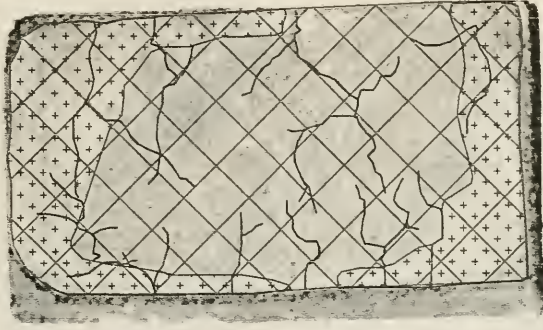




Sample F1  
Treated with 6 cc. 10% Gelatin in  
Water



Sample P1  
Treated with 7 cc. 10% Gelatin in  
Water



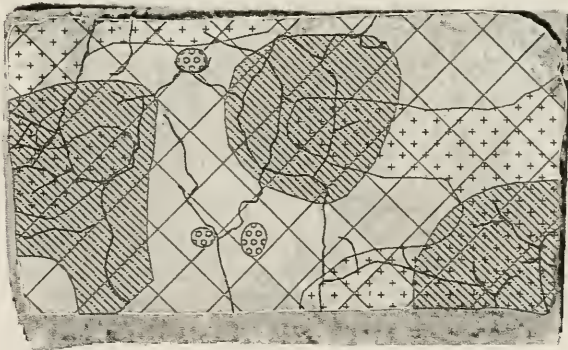
Sample X1  
Treated with 7 cc. 10% Gelatin  
In Water

# KEY

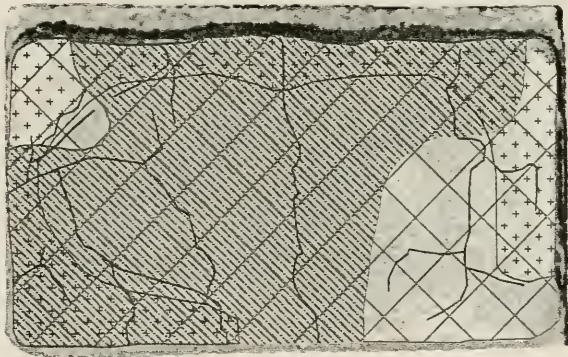
	Plaster Outline		Blisters		Water Damage
	Cracks		Interfacial Delamination		Loss
	Salts		Substrate Detachment		Treated Area



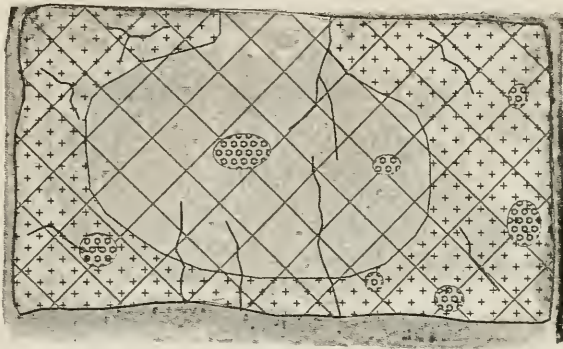




Sample Gi  
Treated with 7 cc. 10% Gelatin &  
10% Glycerine in Water



Sample Zi  
Treated with 6 cc. 10% Gelatin &  
10% Glycerine in Water

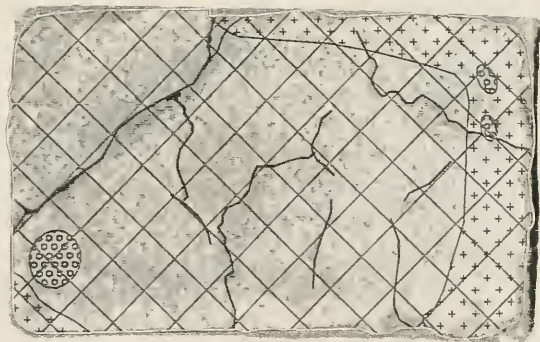


Sample Di  
Treated with 7 cc. 10% Gelatin  
& 10% Glycerine in Water

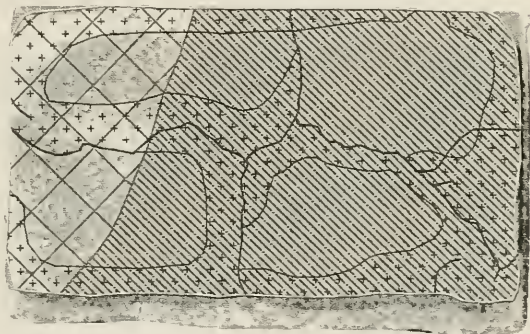
# KEY

	Plaster Outline		Water Damage
	Cracks		Loss
	Salts		Interfacial Delamination
			Substrate Detachment
			Treated Area





Sample I:  
Treated with 5 cc. 10% E-330 in  
Water



Sample R1:  
Treated with 9 cc. 10% E-330 in  
Water

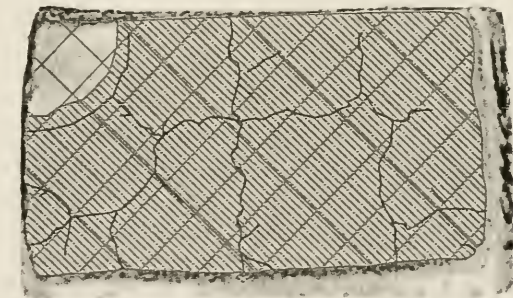


Sample AB:  
Treated with 9 cc. 10% E-330 in  
Water

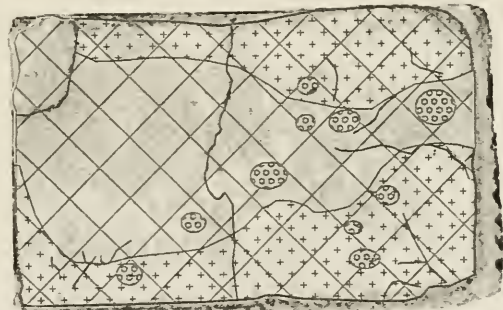
# KEY

	Plaster Outline		Blisters		Water Damage
	Cracks		Interfacial Delamination		Loss
	Salts		Substrate Detachment		Treated Area

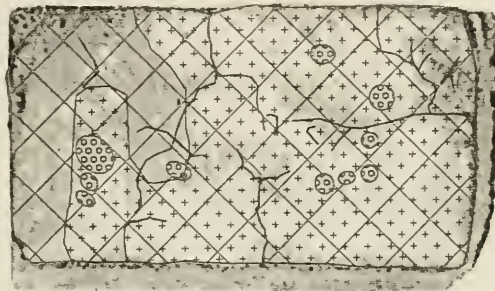




Sample C:  
Treated with 21 cc. 10%  
MC-1834 in Water

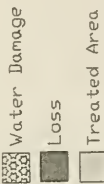
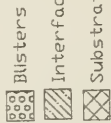
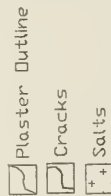


Sample K:  
Treated with 7 cc. 10%  
MC-1834 in Water



Sample U:  
Treated with 7 cc. 10%  
MC-1834 in Water

# KEY





## **APPENDIX P:**

### **Rohm and Haas Product Performance Specification Sheets**







## **RHOPLEX® E-330**

Cement Mortar Modifier

Introduction

Performance Advantages

Physical & Chemical Properties

Suggested Starting Formulations for RHOPLEX® E-330

Formulation Guide with RHOPLEX® E-330

Variables Affecting Cement Mortar Properties Using RHOPLEX® E-330

Safe Handling Information

## **RHOPLEX® E-330**

Cement Mortar Modifier

RHOPLEX® E-330 is a water dispersion of an acrylic polymer specifically designed for modifying Portland cement compositions. Important application areas include patching and resurfacing, floor underlayments, terrazzo flooring, spray and fill coats, precast architectural building panels, stucco, industrial cement floors, and highway and bridge deck repair. Additional information on cement modifiers is available in the technical notes for RHOPLEX® MC-76, RHOPLEX® MC-1834, and DRYCRYL® DP-2903

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### **PERFORMANCE ADVANTAGES**

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- **Durability and Strength:** Cement mortars modified with RHOPLEX® E-330 are hard, tough, and durable. Compared with unmodified mortars, polymer-modified mortars have superior flexural, adhesive, and impact strengths, as well as excellent abrasion resistance. They are especially useful where thin sections are desirable and where excessive vibration and heavy traffic are encountered.
- **Adhesion:** RHOPLEX® E-330-modified cement mortars have excellent adhesion to a variety of surfaces, such as concrete, masonry, brick, wood, and metals.
- **Resistance Properties:** Cement mortars prepared with RHOPLEX® E-330 are resistant to many industrial chemicals and have excellent resistance to ultraviolet light and heat. They dry to a uniform color with no tendency



toward yellowing or discoloration.

- **Curing Advantages:** For optimum physical properties, cement mortars modified with RHOPLEX® E-330 should be air-cured at ambient temperature and relative humidity. Unlike unmodified mortars, which require laborious moist curing conditions for optimum strength properties, polymer-modified mortars should not be cured under these conditions.
- **Storage Advantages:** RHOPLEX® E-330 emulsion is sediment-free and stable to a minimum of five cycles of freezing at -15°C and thawing at 25°C.

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## TYPICAL PHYSICAL PROPERTIES

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Appearance	White, milky liquid
Solids content, %	47 ± 0.5%
pH, when packed	9.3 to 10.2
Specific gravity	1.059
Pounds per gallon	8.8
Freeze/thaw stability	5 cycles
Minimum film-formation temperature	10°C to 12°C

---

RHOPLEX® E-330 is stable for a minimum of five cycles of freezing at -15°C and thawing at 25°C. However, in cold weather or after prolonged storage, the emulsion should be thoroughly stirred prior to use to ensure a completely homogeneous mixture.

Suggested starting point formulations, test results, and other technical information for RHOPLEX® E-330 are noted in the Technical Data Sheets for this product.

## SUGGESTED STARTING FORMULATIONS FOR RHOPLEX® E-330

Depending upon the particular application involved, a variety of cement mortar formulations shown below are useful as starting point systems.

The quantities of water cited in the formulations below should be considered as rough guides. Exact amounts depend on the type and brand of cement, particle size and moisture content of the sand, and on the other agents used in the mortar mix. Increasing amounts of RHOPLEX E-330 used in the cement mortar require decreasing amounts of water for a suitable workable consistency.

To prepare RHOPLEX E-330-modified mortar for evaluation, thoroughly premix the sand and cement. The RHOPLEX E-330, water and antifoamer should be blended together and added to the premixed sand and cement. The entire composition is mixed thoroughly for about two to four minutes. When preparing mortars for application in the field, a portion of the water should be withheld and added gradually to the modified mortar mixture until the desired consistency is obtained. This is necessary to avoid overly fluid compositions in those cases where the sand is used in high moisture content or where other variables affect the amount of water to be used.

Prior to testing, all mortar specimens were cured for 28 days. Unmodified mortars



were prepared by both air-cured and wet-cured procedures, whereas the polymer-modified samples were all air-cured. The conditions employed for air and wet-curing are as follows:

#### AIR-CURING CONDITIONS

- 28 days at 25°C and 50% relative humidity

#### WET-CURING CONDITIONS

- 1 day at 25°C and 90% relative humidity
- 6 days water immersion at 25°C
- 7 days at 25°C and 50% relative humidity
- 7 days water immersion at 25°C
- 7 days at 25°C and 50% relative humidity

### SUGGESTED STARTING FORMULATIONS

Ratio of Polymer Solids to Cement(a)	0.00	0.10	0.15	0.20
Material	Weight	Weight	Weight	Weight
Sand	300.00	300.00	300.00	300.00
Portland Cement (Type 1)	100.00	100.00	100.00	100.00
RHOPLEX® E-330 (47% Solids)	0.00	21.00	32.00	42.00
Defoamer(b)	0.00	0.10(C)	0.15(C)	0.20(C)
Water	48.00	29.00	20.00	11.00
Ratio of Water to Cement(a)	0.48	0.40	0.37	0.35
Ratio of Sand to Cement(a)	3.00	3.00	3.00	3.00

(a) By weight  
 (b) Recommended Defoamers: Nopco NXZ (100% active) - Henkel Corporation, Ambler, PA 19002-3491 GE Antifoam #60 (30% active) - General Electric Co., Silicone Products Dept., Waterford, NY  
 (c) Suggested minimum of 1% based on polymer solids using 100% active defoamer. More may be added, if necessary, to maximize wet density.

### FORMULATION GUIDE WITH RHOPLEX® E-330

#### ACRYLIC SPACKLE COMPOUND FORMULATION (JK-789C)

	Pounds
RHOPLEX E-330(47%)	228
TAMOL® 850(30%)	2
Ethylene glycol	48
ACRYSOL® ASE-60(28%)	4
#8 Marble dust(Georgia Marble Co., Tate,Ga.)	980
Titanium dioxide(Ti-Pure R-901)	12
Total	1274



were prepared by both air-cured and wet-cured procedures, whereas the polymer-modified samples were all air-cured. The conditions employed for air and wet-curing are as follows:

#### AIR-CURING CONDITIONS

- 28 days at 25°C and 50% relative humidity

#### WET-CURING CONDITIONS

- 1 day at 25°C and 90% relative humidity
- 6 days water immersion at 25°C
- 7 days at 25°C and 50% relative humidity
- 7 days water immersion at 25°C
- 7 days at 25°C and 50% relative humidity

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Ratio of Polymer Solids to Cement(a)	0.00	0.10	0.15	0.20
Material	Weight	Weight	Weight	Weight
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Portland Cement (Type 1)	100.00	100.00	100.00	100.00
RHOPLEX® E-330 (47% Solids)	0.00	21.00	32.00	42.00
Defoamer(b)	0.00	0.10(C)	0.15(C)	0.20(C)
Water	48.00	29.00	20.00	11.00
Ratio of Water to Cement(a)	0.48	0.40	0.37	0.35
Ratio of Sand to Cement(a)	3.00	3.00	3.00	3.00

(a) By weight

(b) Recommended Defoamers: Nopco NXZ (100% active) - Henkel Corporation, Ambler, PA 19002-3491 GE Antifoam #60 (30% active) - General Electric Co., Silicone Products Dept., Waterford, NY

(c) Suggested minimum of 1% based on polymer solids using 100% active defoamer. More may be added, if necessary, to maximize wet density.

### FORMULATION GUIDE WITH RHOPLEX® E-330

#### ACRYLIC SPACKLE COMPOUND FORMULATION (JK-789C)

	Pounds
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Ethylene glycol	48
ACRYSOL® ASE-60(28%)	4
#8 Marble dust(Georgia Marble Co., Tate, Ga.)	980
Titanium dioxide(Ti-Pure R-901)	12
Total	1274





### Mixing Procedure

Using a double-bladed sigma-type mixer:

- 1) Premix the liquid ingredients and charge about 75% to the mixer.
- 2) Add sufficient #8 Marble dust and TiO<sub>2</sub> to make a thick paste (approximately 70% of the dry ingredients) while mixing.
- 3) Add the remainder of the liquids and mix thoroughly.
- 4) Add the remainder of the dry ingredients.
- 5) Cover mixer tightly and mix for about one hour.
- 6) About five minutes before the completion of the batch, add 0.3 to 0.4 lb of Nopco NXZ defoamer (0.25% based on polymer solids).

## VARIABLES AFFECTING CEMENT MORTAR PROPERTIES USING RHOPLEX® E-330

Polymer Modification In general, cement mortars modified with RHOPLEX E-330 and air-cured have superior flexural, shear bond adhesion, and impact strengths when compared to moist cured, unmodified cement mortars. In addition, polymer modification results in improved abrasion resistance and comparable tensile and compressive strengths when compared to unmodified mortars. This information is presented in the table below and the figures represent average values of a large number of samples tested.

### PHYSICAL STRENGTH PROPERTIES OF PORTLAND CEMENT MORTAR

Ratio of RHOPLEX® E-330 Polymer	0.00	0.10	0.15	0.20
Ratio of water to cement(1)	0.48	0.40	0.37	0.35
Tensile Strength, psi				
28 day air-cure	235	530	615	855
28 day wet-cure	535			
28 day air-cure +7 day water soak	310	330	350	490
Compressive Strength, psi				
28 day air-cure	2390	5450	5715	5690
28 day wet-cure	5795			
28 day air-cure + 7 day water soak	4420	4700	5125	5460
Flexural Strength, psi				
28 day air-cure	610	1355	1585	1835
28 day wet-cure	1070			
28 day air-cure + 7 day water soak	735	950	1020	1050
Shear Bond Adhesion, psi(2)				



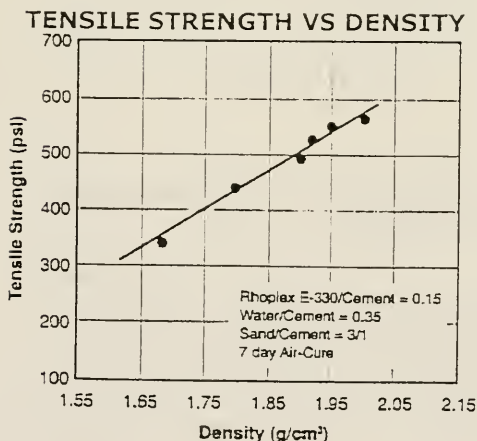
28 day wet-cure	185(A)			
28 day air-cure + 7 day water soak	140(A)	290(C)	300(C)	330(C)
Impact Strength, in/lb				
28 day air-cure	6	12	16	22
28 day wet-cure	7			
28 day air-cure + 7 day water soak	9	11	13	18
Abrasion Resistance, percent weight loss(3)				
28 day air-cure	23.8	1.70	1.15	1.57
28 day wet-cure	5.07			

(1) The water content of the mortars was adjusted to provide equivalent workability, i.e., the polymer-modified mortars were prepared at water contents necessary to give the same slump diameter which was equivalent in workability to the unmodified mortar controls at 48 percent water content. This procedure is described in the ASTM "Flow Rate" test #C-230-83.  
(2) Adhesive failure indicated by (A). Cohesive failure indicated by (C).  
(3) Lower values indicate better abrasion resistance.

## VARIABLES AFFECTING CEMENT MORTAR PROPERTIES USING RHOPLEX® E-330

### Density

The graph below shows the effect of density variation on the tensile strength of cement mortar modified with RHOPLEX E-330. As the density increases, so does the tensile strength. Similarly, one can improve the compressive, flexural impact and adhesive strengths by increasing the mortar density. Therefore, when modifying cement mortars with RHOPLEX E-330 it is important to minimize the air entrainment due to foaming. By using an appropriate amount of antifoamer it is possible to get high density, polymer-modified mortars with excellent strength properties. In general, the wet density of a latex-modified cement mortar should be at least the same as unmodified mortar. In most cases the wet density will be 2.0 g/cm<sup>3</sup> or higher.



## VARIABLES AFFECTING CEMENT MORTAR PROPERTIES USING RHOPLEX® E-330

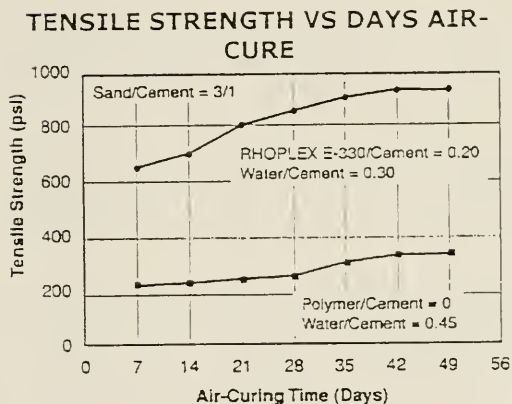


### Length of Cure

All cement mortars continue to cure with the passage of time. It is believed that it takes about 28 days for both a polymer-modified and an unmodified cement mortar to obtain approximately 90 percent of their ultimate physical properties. The graph below shows the increase in tensile strengths of RHOPLEX E-330-modified and unmodified mortars as a function of time. Similar behavior also occurs in other physical strength properties.

### Curing Conditions

To obtain maximum physical strength properties, RHOPLEX E-330-modified cement mortars should be air-cured at ambient temperature and relative humidity, avoiding the use of moist curing techniques, a laborious procedure used with unmodified mortars to obtain optimum properties.



## VARIABLES AFFECTING CEMENT MORTAR PROPERTIES USING RHOPLEX® E-330

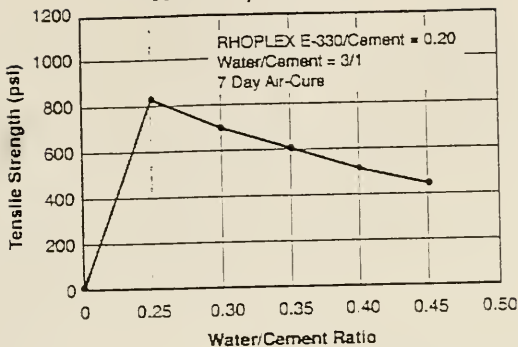
### Water Level

The amount of water that should be used in a cement mortar depends on the type and brand of cement used, the particle size and moisture content of the sand used, the additional ingredients of the mortar mix, and the working consistency desired for the particular application. As a general rule, water should be held to the lowest amount needed to achieve a suitable working consistency. By using a minimum amount of water, maximum strength properties are obtained.

The effect of the water/cement ratio on tensile strength at a constant polymer level is shown in the graph below:



## TENSILE STRENGTH VS WATER/CEMENT



## SAFE HANDLING INFORMATION

Animal toxicity screening tests conducted on closely related analogs of RHOPLEX® E-330 suggest that this product should be essentially nontoxic by single acute oral or dermal exposure and that it may also be a mild to moderate skin and eye irritant. In addition, many of the components of cement used in conjunction with RHOPLEX E-330 may also possess significant skin and eye irritation potential.

The Rohm and Haas Company maintains comprehensive and up-to-date Material Safety Data Sheets (MSDS) on all of its products. These sheets contain pertinent information that you may need to protect your employees and customers against any known health or safety hazards associated with our products.

The Rohm and Haas Company recommends that you obtain copies of our Material Safety Data Sheets from your local Rohm and Haas representative on each of our products prior to its use in your facilities. We also suggest that you contact your supplier of other materials recommended for use with our products for appropriate health and safety precautions prior to their use.

*ACRYSOL®, RHOPLEX, and TAMOL® are registered trademarks of Rohm and Haas Company or of its subsidiaries or affiliates, and are intended to designate goods marketed in North and South America; the same goods may be marketed in other countries, generally under the Company trademark designations.*

*These suggestions and data are based on information we believe to be reliable. They are offered in good faith, but without guarantee, as conditions and methods of use of our products are beyond our control. We recommend that the prospective user determine the suitability of our materials and suggestions before adopting them on a commercial basis.*

*Suggestions for uses of our products or the inclusion of descriptive material from patents and the citation of specific patents in this publication should not be understood as recommending the use of our products in violation of any patent or as permission or license to use any patents of the Rohm and Haas Company.*





## **APPENDIX Q:**

### **Rohm and Haas Product Materials Safety Data Sheets**



# MATERIAL SAFETY DATA SHEET

## Rohm and Haas Company

### 1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

RHOPLEX™ E-330 Emulsion

Product Code : 66580  
Key : 905688-9

MSDS Date : 08/05/99

RHOPLEX™ is a trademark of Rohm and Haas Company or one of its subsidiaries or affiliates

*Italics denote a revision from previous MSDS.*

COMPANY IDENTIFICATION	EMERGENCY TELEPHONE NUMBERS
Rohm and Haas Company 100 Independence Mall West Philadelphia, Pa 19106-2399	HEALTH EMERGENCY : 215-592-3000 SPILL EMERGENCY : 215-592-3000 CHEMTREC : 800-424-9300

### 2. COMPOSITION/INFORMATION ON INGREDIENTS

No		CAS REG NO	WEIGHT (%)
1	<i>P(BA/MMA)</i>	25852-37-3	46-48
2	<i>Residual monomers</i>	<i>Not Required</i>	<0.05
3	<i>Aqua ammonia</i>	1336-21-6	0.3 MAX
4	<i>Water</i>	7732-18-5	52-54

*Polymeric description(s) presented in this section are the U.S. Toxic Substances Control Act (TSCA) definitions.*

See Section 8, Exposure Controls / Personal Protection

### 3. HAZARDS IDENTIFICATION



## Primary Routes of Exposure

*Inhalation*

*Eye Contact*

*Skin Contact*

### Inhalation

*Inhalation of vapor or mist can cause the following:*

- headache - nausea - irritation of nose, throat, and lungs

### Eye Contact

*Direct contact with material can cause the following:*

- slight irritation

### Skin Contact

Prolonged or repeated skin contact can cause the following:

- slight skin irritation

## 4. FIRST AID MEASURES

### Inhalation

Move subject to fresh air.

### Eye Contact

*Flush eyes with water. Consult a physician if irritation persists.*

### Skin Contact

Wash affected skin areas thoroughly with soap and water. Consult a physician if irritation persists.

### Ingestion

*If swallowed, give 2 glasses of water to drink. Never give anything by mouth to an unconscious person. Consult a physician.*

## 5. FIRE FIGHTING MEASURES



Flash Point	Noncombustible
Auto-ignition Temperature	Not Applicable
Lower Explosive Limit	Not Applicable
Upper Explosive Limit	Not Applicable

### Unusual Hazards

*Material can splatter above 100C/212F. Dried product can burn.*

### Extinguishing Agents

Use extinguishing media appropriate for surrounding fire.

### Personal Protective Equipment

*Wear self-contained breathing apparatus (pressure-demand NIOSH approved or equivalent) and full protective gear.*

## 6. ACCIDENTAL RELEASE MEASURES

### Personal Protection

*Appropriate protective equipment must be worn when handling a spill of this material. See SECTION 8, Exposure Controls/Personal Protection, for recommendations. If exposed to material during clean-up operations, see SECTION 4, First Aid Measures, for actions to follow.*

### Procedures

*Contain spills immediately with inert materials (e.g. sand, earth). Floor may be slippery; use care to avoid falling. Transfer liquids and solid diking material to separate suitable containers for recovery or disposal. Keep spectators away.*

*CAUTION: Keep spills and cleaning runoff out of municipal sewers and open bodies of water.*

## 7. HANDLING AND STORAGE

### Storage Conditions

*Keep from freezing; material may coagulate. The minimum recommended storage temperature for this material is 1C/34F. The maximum recommended storage temperature for this material is 60C/140F.*

### Handling Procedures

*Monomer vapors can be evolved when material is heated during processing operations. See*





## 8. EXPOSURE CONTROLS/PERSONAL PROTECTION

### Exposure Limit Information

No		CAS REG NO	WEIGHT (%)
1	P(BA/MMA)	25852-37-3	46-48
2	Residual monomers	Not Required	<0.05
3	Aqua ammonia	1336-21-6	0.3 MAX
4	Water	7732-18-5	52-54

Comp.		ROHM AND HAAS		OSHA		ACGIH	
No.	Units	TWA	STEL	TWA	STEL	TWA	STEL
1		None	None	None	None	None	None
2		a	a	a	a	a	a
3	ppm	25 b	35 b	None	35 b	25 b	35 b
4		None	None	None	None	None	None

a Not Required

b As Ammonia

The CAS # of the polymer component(s) disclosed above provides information about the major monomers used to manufacture the product. Trace levels of these monomers may be present.

### Respiratory Protection

A respiratory protection program meeting OSHA 1910.134 and ANSI Z88.2 requirements must be followed whenever workplace conditions warrant a respirator's use. None required if airborne concentrations are maintained below the exposure limit listed in 'Exposure Limit Information'. For airborne concentrations up to 10 times the exposure limit, wear a NIOSH approved (or equivalent) half-mask, air-purifying respirator. Air-purifying respirators should be equipped with NIOSH approved (or equivalent) cartridges for protection against ammonia and methylamine and filters for protection against dusts and mists.

### Eye Protection

Use safety glasses with side shields (ANSI Z87.1 or approved equivalent). Eye protection worn must be compatible with respiratory protection system employed.



## Hand Protection

The glove(s) listed below may provide protection against permeation. Gloves of other chemically resistant materials may not provide adequate protection:

- Neoprene

## Engineering Controls (Ventilation)

*Use local exhaust ventilation with a minimum capture velocity of 100 ft/min. (0.5 m/sec.) at the point of vapor evolution. Refer to the current edition of **Industrial Ventilation: A Manual of Recommended Practice** published by the American Conference of Governmental Industrial Hygienists for information on the design, installation, use, and maintenance of exhaust systems.*

## Other Protective Equipment

Facilities storing or utilizing this material should be equipped with an eyewash facility.

# 9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance	Milky
Color	White
State	Liquid
Odor Characteristic	Ammonia odor
pH	9.3 to 10.2
Viscosity	5 to 55 CPS
Specific Gravity (Water = 1)	1.0 to 1.2
Vapor Density (Air = 1)	< 1 Water
Vapor Pressure	17 mm Hg @ 20°C/68°F Water
Melting Point	0°C/32°F Water
Boiling Point	100°C/212°F Water
Solubility in Water	Dilutable
Percent Volatility	52 to 54 % Water
Evaporation Rate (BAc = 1)	< 1 Water

See Section 5, Fire Fighting Measures

# 10. STABILITY AND REACTIVITY



## Instability

*This material is considered stable. However, avoid temperatures above 177C/350F, the onset of polymer decomposition. Thermal decomposition is dependent on time and temperature.*

## Hazardous Decomposition Products

Thermal decomposition may yield acrylic monomers.

## Hazardous Polymerization

Product will not undergo polymerization.

## Incompatibility

There are no known materials which are incompatible with this product.

# 11. TOXICOLOGICAL INFORMATION

## Acute Data

*No toxicity data are available for this material.*

*The information shown in SECTION 3, Hazards Identification, is based on the toxicity profiles for a number of acrylic emulsions that are compositionally similar to this product. Typical data are:*

*Oral LD50 - rat: >5000 mg/kg*

*Dermal LD50 - rabbit: >5000 mg/kg*

*Skin irritation - rabbit: practically non-irritating*

*Eye irritation - rabbit: inconsequential irritation*

# 12. ECOLOGICAL INFORMATION

*No data are available for this material. The information shown is based on profiles of compositionally similar materials.*

*Inherent Biodegradability (OECD 302 B): this type of product is not biodegradable but readily bioeliminable (non-inhibiting)*

*Activated Sludge Respiratory Inhibition (OECD 209): >100 mg/l (non-inhibiting)*

## Environmental Toxicity

*The Environmental Toxicity data are for a compositionally similar material.*

*Algae (Selenastrum capricornutum), 72 Hour EC50: > 100 ppm*

*Daphnia Magna, 48 Hour EC50: >100 ppm*



### 13. DISPOSAL CONSIDERATIONS

#### Procedure

*Coagulate the emulsion by the stepwise addition of ferric chloride and lime. Remove the clear supernatant and flush to a chemical sewer.*

*Landfill or incinerate remaining solids in accordance with local, state and federal regulations.*

### 14. TRANSPORT INFORMATION

US DOT Hazard Class

NONREGULATED

### 15. REGULATORY INFORMATION

#### Workplace Classification

This product is considered non-hazardous under the OSHA Hazard Communication Standard (29CFR 1910.1200).

This product is not a 'controlled product' under the Canadian Workplace Hazardous Materials Information System (WHMIS).

#### SARA TITLE 3: Section 311/312 Categorizations (40CFR 370)

This product is not a hazardous chemical under 29CFR 1910.1200, and therefore is not covered by Title III of SARA.

#### SARA TITLE 3: Section 313 Information (40CFR 372)

*This product does not contain a chemical which is listed in Section 313 at or above de minimis concentrations.*

#### CERCLA Information (40CFR 302.4)

*Releases of this material to air, land, or water are not reportable to the National Response Center under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) or to state and local emergency planning committees under the Superfund Amendments and Reauthorization Act (SARA) Title III Section 304.*





## Waste Classification

*When a decision is made to discard this material as supplied, it does not meet RCRA's characteristic definition of ignitability, corrosivity, or reactivity, and is not listed in 40 CFR 261.33. The toxicity characteristic (TC), however, has not been evaluated by the Toxicity Characteristic Leaching Procedure (TCLP).*

## United States

*All components of this product are in compliance with the inventory listing requirements of the U.S. Toxic Substances Control Act (TSCA) Chemical Substance Inventory.*

## Pennsylvania

*Any material listed as "Not Hazardous" in the CAS REG NO. column of SECTION 2, Composition/Information On Ingredients, of this MSDS is a trade secret under the provisions of the Pennsylvania Worker and Community Right-to-Know Act.*

## 16. OTHER INFORMATION

Rohm and Haas Hazard Rating		Scale
Toxicity	1	4=EXTREME
Fire	0	3=HIGH
Reactivity	0	2=MODERATE
Special	-	1=SLIGHT
		0=INSIGNIFICANT

Ratings are based on Rohm and Haas guidelines,  
and are intended for internal use.

## HMIS Hazard Ratings

*HMIS Hazard Ratings: HEALTH = 1, FLAMMABILITY = 0, REACTIVITY = 0.*

*PERSONAL PROTECTION: See Section 8, Exposure*

*Controls/Personal Protection for recommended*

*handling of material as supplied; check with*

*supervisor for your actual use condition.*

*Scale: 0 = Minimal, 1 = Slight, 2 = Moderate, 3 = Serious, 4 = Severe*

*\* = Chronic Effects (See Section 3, Hazards Identification)*

*HMIS is a registered trademark of the National Paint and Coatings Association.*



## **APPENDIX R:**

### **Select American Society for Test Methods Procedures**



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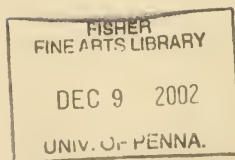
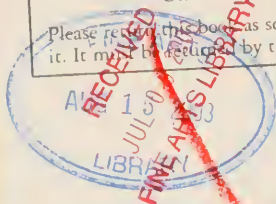




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